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THE INFLUENCE OF PHYSICAL PROPERTIES
ON BLACK POWDER COMBUSTION

Ronald A. Sasse'

March 1981

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
BALLISTIC RESEARCH LABORATORY
ABERDEEN PROVING GROUND, MARYLAND

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It has been observed that black powder made from ingredients of the same lot and fabricated using the same manufacturing procedure results in material exhibiting different burning rates. The current study was undertaken to determine if the physical properties of black powder affected burning rate and establish if these properties varied from one production batch to another.

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20. ABSTRACT: (Continued)

Physical properties of black powder were measured including surface area, pore size distribution, and internal free volume. Black powder derived from oak and maple charcoals was compared, and the effects of processing the material by a jet mill or ball mill were contrasted. Scanning electron microscopy revealed that applied pressure, a manufacturing step used in making black powder, induced considerable plastic flow that resulted in a fused conglomerate, cohesive mass containing interconnecting passageways of about 0.1 micron diameter. Density, free volume, and surface area were found to be functions of burning rate. Based on these relationships, the conclusion was offered that the degree of openness of a grain of black powder is a significant factor affecting its burning rate.

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I. INTRODUCTION

Black powder was made in production mills in Europe as early as 1340 in Augsburg, 1344 in Spandau and 1348 in Legnica. The process, traceable to the early B.C. effort of the Chinese, evolved as an "art form" having historical rather than scientific origins. The development of the craft has been reconstructed by various authors and excellent accounts have been given by Urbanski[1] and Marshall.[2] The subject has been reviewed by Fedoroff and Sheffield[3] who provide numerous references.

In essence, black powder has been made by grinding potassium nitrate, sulfur and charcoal into a very fine powder. This material is transformed into a cohesive conglomerate mass by either forming a paste which is forced through and divided by a screen, or the material is pressed into a cake. Both forms are subsequently broken into pieces. The small lumps or grains of black powder are then coated with graphite to retard the absorption of moisture and to prevent the grains from adhering. The grains are then screened to a particular size. This process has been standardized to some degree, in accordance with prevailing technology, but the operator adds water, adjusts temperature, selects grinding time, or changes compression pressure based on personal experience which is guided by individual judgment of color, odor, or general perception of the condition of black powder as it undergoes its various transformations. Such judgments are exchanged from experienced operator to journeyman.

Although the production of black powder was as great as 10.6 million pounds[4] during World War I, the advent of smokeless powder diminished demand. This fact together with the inherent dangers of production, marked by occasional explosions, has resulted in closing most manufacturing facilities in North America. Because black powder still plays an important role in fuses and ignition devices the U. S. Army Corps of Engineers has built an Ammunition Plant in Charlestown, IN and equipment was installed by ICI Americas, Inc. In designing the plant every effort was made to modernize the black powder manufacturing process and to make it as safe as possible. In the planning stage various studies were

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[1] Tadeusz Urbanski, *Chemistry and Technology of Explosives,* Vol 3, *Pergamon Press, NY,* pp. 322-346, (1967).

[2] Arthur Marshall, *Explosives,* Sec. Ed. Vol 1, *History and Manufacture,* P. Blakiston's Son and Co., Philadelphia, (1917).

[3] B. T. Fedoroff and O. E. Sheffield, *Encyclopedia of Explosives and Related Items,* Vol 2., PATR 2700, Picatinny Arsenal, Dover, NJ, pp. B165-B178, (1962).

[4] Arthur P. Vangelder and Hugo Schatter, *History of the Explosive Industry in America,* NY Columbia University Press, NY (1927).

conducted to: (1) elucidate and evaluate the ball and wheel mill process, (2) to investigate new techniques and equipment and (3) to compare the ballistic properties of black powder originating from this and other countries. The accumulated knowledge was used as a foundation for the design of the Indiana Plant. This work was reported in part by the Chromalloy Corporation[5] Battelle Memorial Institute,[6] Olin Corporation[7] and ICI Americas, Inc.[8] Battelle[5] published abstracts of 50 articles and 60 patents describing the wheel mill process in fine detail. This process, sometimes referred to as the "standard process", and used in the 1860's can be currently witnessed at the Belin Powder Works in Moosic, PA. The plant was built in 1911-12 and operated for many years by the E. I. du Pont de Nemours & Co. The plant is now owned and operated by GOEX, Inc., and supplies most of the military black powder requirements.

Particle size distributions obtained by "the standard process" were measured, using optical techniques, by Battelle Memorial Institute,[6] as a function of both grinding time and moisture content. These results were compared to black powder made by another technique, the jet mill process, in which material is ground by impaction created by high velocity air streams. The jet mill process was developed by Kjell Lovold[9] and is used in Norway to make black powder. The technique was incorporated in the Indiana Plant. Using the jet mill, particles are generally one third smaller than produced by the wheel mill. Also, J. C. Allen[10] compared the

[5]Chromalloy Corporation, "A Study of Modernized Techniques for the Manufacture of Black Powder", Propellex Chemical Division, Final Report No. DAI-23-072-501-ORD-P-43, Jan 60, Chromalloy Corp., IL.

[6]H. E. Carlton, B. B. Bohrer, and H. Naok, "Battelle Memorial Institute Final Report on Advisory Services on Conceptual Design and Development of New and Improved Processes for the Manufacture of Black Powder", Olin Corporation, Indiana Army Ammunition Plant, 20 Oct 1970, Charlestown, IN.

[7]J. R. Plessinger and L. W. Braniff, "Final Report on Development of Improved Process for the Manufacture of Black Powder", RCS AMURE-109, Olin Corporation, Indiana Army Ammunition Plant, 31 Dec 1971, Charlestown, IN.

[8]Indiana Ammunition Plant, "Black Powder Manufacturing Facility", Vol. 1 and 2, Indiana Army Ammunition Plant, 1975, Charlestown, IN.

[9]Kjell Lovold, U.S. Patent No. 3660546, "Process for the Preparation of Black Powder", 2 May 1972.

[10]J. C. Allen, "The Adequacy of Military Specification MIL-P-223B to Assure Functionally Reliable Black Powder", Report No. ASRSD-QA-A-P-60, Ammunition System Reliability and Safety Div, Product Assurance Directorate, June 1974, Picatinny Arsenal, Dover, NJ.

effects of different milling procedures at various production facilities used in this and other countries.

Other types cf preparation have been attempted which include dissolving sulfur[5] by either carbon disulfide or alcohol and mixing this solution, or slurry, with charcoal and potassium nitrate before evaporating. Another innovation of note was pursued by Voigt[11] in which potassium nitrate was precipitated from solution by adding alcohol in a controlled manner to produce fine particles.

From the extensive background one general observation made in regard to the production of black powder is that the same manufacturing procedure, using the same ingredients, will result in lots exhibiting different burning rates. This has been the experience of GOEX, Inc. where fast and slow lots are blended to obtain a particular result. Although some of the factors which effect burn rate are known, the lot-to-lot variations have not been identified. One attempt to study the effect of small perturbations was supported by the Product Assurance Directorate, under the direction of J. C. Allen[12] where planned and slight deviations from a normal production run were purposefully and systematically introduced in ten lots of black powder made by the Indiana pilot plant. These samples were used to determine what manufacturing parameters are important in effecting th performance and combustion of black powder. These lots were termed ~ "deviant lots" and will be described later. The burning characteristics of these and other standard lots were measured by four different laboratories: ARRADCOM (BRL[13], LCWSL[14]), ICI Americas Corp.[15]

[11] H. William Voigt, Jr., Pell W. Lawrence, and Jean P. Picard, "Process for Preparing Modified Black Powder Pellets", U.S. Patent, 3937771, Feb 10, 1976, see also H. W. Voigt and D. S. Downs, "Simplified Processing of Black Powder and Pyrotechnical Igniter Pills Including Low Residue Versions", Seventh International Pyrotechnics Seminar, Vol. 2, ITT Research Inst., Chicago IL, July 1980.

[12] J. C. Allen, "Concept Scope of Work For MM&TE Project 5764303 Acceptance of Continuously Produced Black Powder", Report No. SARPA-QA-X-10, November 1975, Picatinny Arsenal Dover, NJ.

[13] K. J. White, H. E. Holmes, J. R. Kelso Jr., A. W. Horst, and I. W. May, "Ignition Train and Laboratory Testing of Black Powder", ARRADCOM-BRL Report No. IMR-642, April 1979, APG, MD. See also K. J. White, H. E. Holmes, and J. R. Kelso, "Effect of Black Powder Combustion on High and Low Pressure Igniter Systems", 16th JANNAF Combustion Meeting, CPIA Publication 308, Dec 1979.

[14] L. Shulman, private communication, (data quoted in reference 13), 15 Jan 1978, Picatinny Arsenal, Dover, NJ.

[15] Hugh Fitler, private communication - Draft report, "Acceptance of Continuously Produced Black Powder", 8 March 1979, ICI America Corp., Charlestown, IN.

and Princeton Combustion Research Laboratories.[16] These results were compared by Kevin White[13] and will be presented later; it was concluded that the same trends were observed in each laboratory. Although there were differences in burning rates between various lots, the conclusion was that small perturbations in manufacturing procedures do not account for the difference of a factor of two in the observed flame spread rates. When these lots were compared with some GOEX, Inc. black powder, it was inferred that some other unidentified variable must be operative in these samples.

The performance of black powder, in contrast to its preparation, has been characterized in the classic papers of Noble and Abel[17-18]. Recently, Williams[19] summarized burning characteristics and included several foreign references. Rose[20] studied the performance of black powder made from charcoal produced by various manufacturers and concluded that response could be traced to volatile content; he also presented an excellent review article.[21] Kirshenbaum[22] concluded from thermal DTA analysis that even when volatiles were removed from charcoal the same order of ignition was maintained and some other parameter besides volatiles affected the ignition of black powder. He also found that ignition temperature was not a function of carbon surface area, ash content nor of sulfur content. Further, activation energy was not a function of volatile content. Blackwood and Bowden[23] suggested that sulfur reacts with volatiles as an initiation step but Kirshenbaum showed that all three components, sulfur, potassium nitrate and charcoal, must be

[16] Neale A. Messina, Larry S. Ingram and Martin Summerfield, "Black Powder Quality Assurance Flame Spread Tester", Final Report No. PCRL-TR-78-101, Dec 1978, Princeton Combustion Laboratories Inc., Princeton NJ.

[17] R. A. Noble and F. Abel, Phil. Trans. Roy. Soc., London, Series A Vol. 165, 49-155, (1875).

[18] R. A. Noble and F. Abel, Phil. Trans. Roy. Soc., London, Series A 203-279, (1880).

[19] F. Williams, "The Role of Black Powder in Propelling Charges", Picatinny Arsenal Tech. Report No. 4770, May 1975, Picatinny Arsenal, Dover, NJ.

[20] James E. Rose, "Investigation on Black Powder and Charcoal", IHTR-433, Sept. 1975, Naval Ordnance Station, Indian Head, MD.

[21] James E. Rose, "Black Powder - A Modern Commentary", Proc. of the 10th Symposium on Explosives and Pyrotechnics, 14-16, 1979, Franklin Research Inst. Philadelphia, PA.

[22] Abraham D. Kirshenbaum, Thermochimica Acta, 18, 113, (1977).

[23] J. D. Blackwood and F. P. Bowden, Proc. Roy. Soc., London, A213, 285, (1952).

present to induce a low temperature pre-ignition exotherm. Perhaps one of the most persuasive experiments on the importance of volatiles was that of Blackwood and Bowden in which they dissolved volatiles from charcoal with acetone and applied them to charcoal that was 95 percent carbon. Black powder made from this material had the same behavior as standard powder.

Clearly, volatiles affect burning rate and Hintze[24] shows the relationship. He recommends 82.5 percent carbon content for charcoal and Blackwood and Bowden recommended 70 percent. However, neither work relates these percentages to a standard initial form of water or ash-free basis and it is not clear that the recommendations can be compared directly. Both authors do recommend a rather high volatile content. However, this parameter cannot be the only property that affects burning rate for the deviant lots have diverse burning rates incorporating the same charcoal.

In addition to the affect of volatiles, Shulman[25,26] studied the influence of small amounts of water, to two percent on the burning rate of black powder; this study was expanded by Plessinger.[7] These investigations quantify a well-known parameter.

From this discussion it is seen that the properties of charcoal are important, but the exact nature of the requirements necessary to make a good ballistic product are elusive. Present practice is that charcoal be made from porous light wood, maple being currently selected, and be of low ash content. No specification exists citing volatile content but current practice is that it be 20-30%. This specification is broad in that it requires charcoal which results in a proper performing black powder. The specification does not cite the wood, particulars relating to distillation, or physical properties. For black powder the amount of potassium nitrate, sulfur, and charcoal is given but compaction pressure is not. Only a density range for black powder is given. Such criteria result in a large latitude in allowable variance in the materials used to make black powder.

It appeared to this author that rather large variations in materials or in preparation of black powder resulted in rather small but measurable

[24] Woldemar Hintze, *Explosivstoffe*, 2, 41, (1968).

[25] L. Shulman, C. Lenchitz, L. Bottei, R. Young and P. Casiano, "An Analysis of the Role of Moisture, Grain Size, and Surface Glaze on the Combustion and Functioning of Black Powder", Picatinny Arsenal Report No. 75-FR-G-B-15, Oct. 1975, Picatinny Arsenal, Dover, NJ.

[26] L. Shulman, R. Young, E. Hayes and C. Lenchitz, "The Ignition Characteristics of Black Powder", Picatinny Arsenal Technical Report No. 1805, Sept. 1967, Picatinny Arsenal, Dover, NJ.

variations in burning rate; lot-to-lot differences, using the same materials and techniques, was a dominant feature. It was for these reasons that another sensitive parameter was sought and a candidate hypothesis was that the degree of openness of a grain could influence burning rate. Soon the study evolved into an investigation of the physical properties of black powder and their relation to burning rate.

In an attempt to characterize black powder, scanning electron microscope photographs were obtained which showed extensive fusing of material. This immediately suggested that compaction studies be performed on pure ingredients to determine what material flowed and the functional relationship between flow and applied pressure. In addition the free volume was measured by mercury intrusion techniques. Further, the internal surface area was obtained from the Brunauer, Emmett, and Teller (B.E.T.) gas absorption technique. From these several measurements a physical description of black powder developed.

II. EXPERIMENTAL

A. Black Powder Samples

Deviant lots, mentioned earlier, were chosen for study as they were: (1) well characterized ballistically by four laboratories, (2) made from ingredients supplied by the same manufacturer, (3) fabricated by the jet mill process, and (4) representative pilot plant samples typical of future production techniques. Such samples were compared to black powder made by the "standard" wheel mill process at GOEX, Inc. The deviant lots were made from oak charcoal and GOEX, Inc., used maple. Equivalent compaction pressures were used by both manufacturers.

Due to the limited capacity of the Indiana pilot plant, their jet-milled meal was moisturized in a wheel mill at Picatinny Arsenal and pressurized at GOEX, Inc., to not less than 3500 psi, or 246 kg/cm^2. Fabrication has been described in detail[15] and composition given in Table 1. For this table the carbon content has been recalculated on a moisture and ash-free basis. Also to obtain the high and low carbon content charcoals, three different lots of oak charcoal were used. The high carbon content charcoal was but eight percent greater than that normally used and the low carbon material was 17 percent below standard. The spread in these values is significant but small. Such samples were compared to GOEX, Inc., and CIL black powder produced by the standard process and made with maple charcoal. The burning rate data from various laboratories ARRADCOM (BRL,[13] LCWSL[14]), and Princeton Combustion Research Laboratories[16] are compared in Figure 1 reproduced from reference 13. The results are normalized to the burning rate data for a standard GOEX, Inc., lot, 75-44.

TABLE 1. DEVIANT BLACK POWDER LOTS

Lot Number	Description*	Density** g-cm-3	KNO3 %	S %	Charcoal %	Flame Spread*** ms-1
1	High KNO3	1.86	78.01	8.50	11.95	0.32
2	Low KNO3	1.74	73.00	10.40	16.30	0.50
3	Poor Agglomeration of Ingredients	1.77	75.95	9.24	13.0?	0.41
4	High Density	1.80	74.81	9.29	16.05	0.3?
5	Low Density	1.67	74.94	9.07	14.4?	0.43
6	High Glaze - 0.2%	1.78	74.88	9.23	14.08	0.39
7	High Carbon - 79.7%	1.67	74.52	10.13	15.21	0.36
8	Low Carbon - 81.6%	1.70	75.00	10.23	13.86	0.38
9	Large Particle Size - avg over 25 µ	1.70	72.50	10.97	13.59	0.34
10	Small Particle Size - avg over 10 µ	1.63	74.60	10.38	14.02	0.51
11	GOEX - Lot 75-44 - non-standard glaze	1.67	74.29	10.06	15.56	0.60
12	GOEX - Lot 75-44 - standard glaze	1.72	74.05	10.18	15.33	0.53
	GOEX - Lot 75-61					0.57
	GOEX - Lot 75-2					0.60
	CIL-7-11					0.49

* Oak charcoal in lots 1-7 and 9-10 are 73.8% carbon on an ash and moisture free basis.

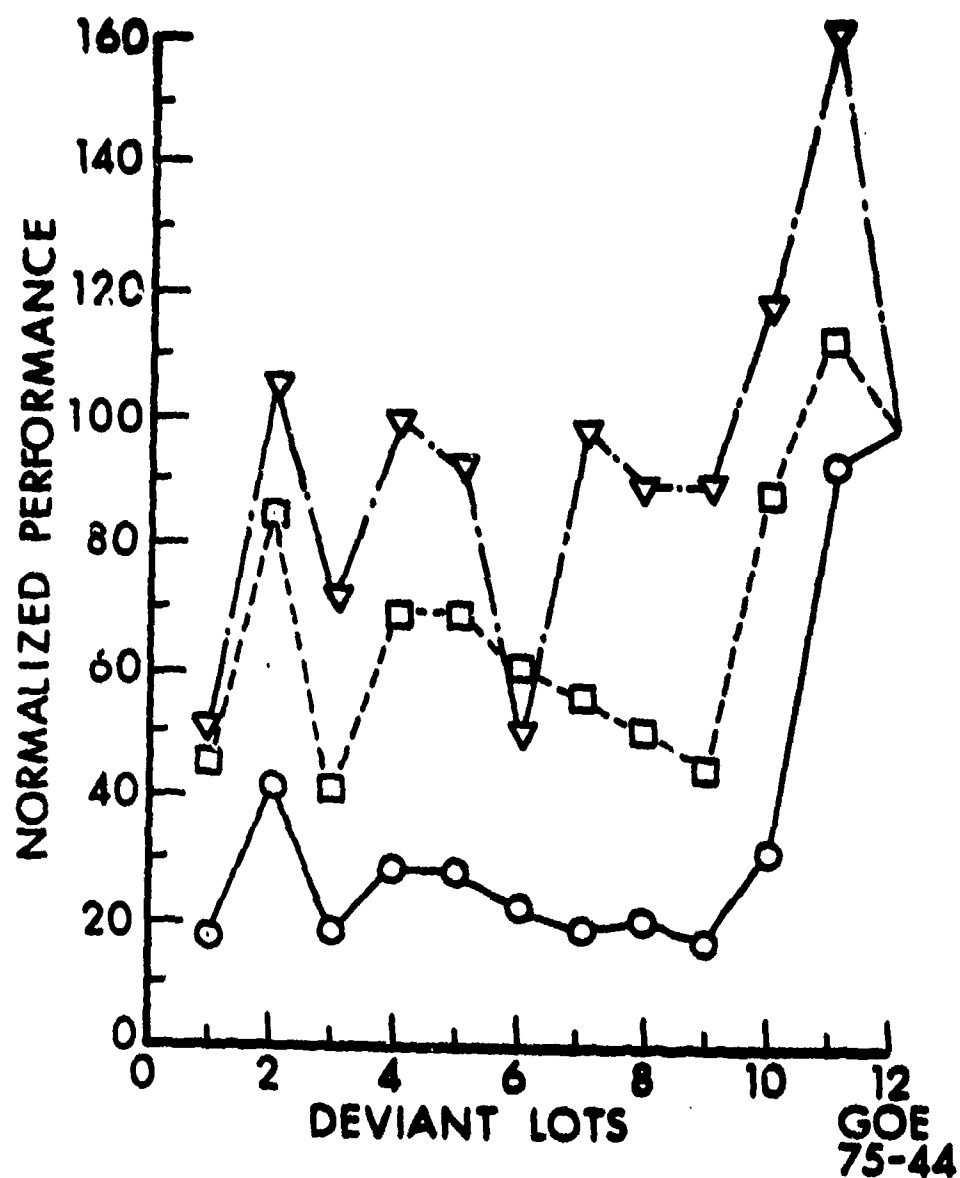** Bulk density

*** Open air flame spread values (ref 13).

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Figure 1. Comparison of burning rates as measured by flame spread, O (ref. 16); closed bomb, □ (ref. 14); and open air flame spread, △ (ref. 13).

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B. Scanning Electron Microscopy of Black Powder and Charcoal

S.E.M. photographs of black powder were obtained at two magnifications, 300 and 1000X, by John Gavel of the Analytical and Physical Measurements Services Section, E.I. du Pont de Nemours & Co., Wilmington, DE. Class one grains, those passing through a number 8 but not a number 4 sieve, were examined. They were cleaved in half by breaking the grains, much like snapping a twig, to insure that no tool smeared the surface. The new surface was coated with a 20 nm gold and palladium film; 30 keV electrons were used for analysis.

Another imaging technique was attempted using an electron microprobe, model M5-64 made by the Acton Laboratories Inc., of Acton, MA. In these experiments the sample was moved normal to the oscillating beam by a motor drive. The experiments were performed by Ralph Benck of the Penetration Mechanics Branch (PMB) of BRL. Both sulfur and potassium x-ray maps were obtained simultaneously using two detectors. Benck also analyzed the charcoal ash, from charcoal extracted from lots 1 and 11.

Other S.E.M. photographs of oak and maple charcoals were taken at the BRL by Gould Gibbons Jr., of the Explosive Effects Branch (EEB), using an International Scientific Corp. S.E.M. model ISI-40 made in Avon, CT. Oak charcoal was evaluated as received from the Indiana Ammunition Plant; however, it was pre-screened. Charcoal passing through an 80 but held on a 100 mesh screen was examined. The maple charcoal was of a larger particle size passing through a 10 but not a 20 mesh screen and, therefore, it was ground in a mortar and pestle to obtain particles of the same size as the oak. Both samples were obtained by the Indiana group from the Hardwood Charcoal Co. located in Steelville, MO. S.E.M. views were obtained at 100, 150, 500 and 2000X.

In addition, oak charcoal was extracted from lot 1 and maple from lot 11 black powder samples. They were extracted with water and carbon disulfide. For lot 11 magnifications of 150, 350, 750, and 1500X were employed; similar values were used for lot 1 and they were: 100, 250, 500, and 1000X.

C. Compression of Sulfur, Potassium Nitrate and Charcoal

Pure sulfur, potassium nitrate, and charcoal were each ground to a fine powder using a mortar and pestle. Samples between 1 and 2 grams were loaded into a Perkin Elmer Corp. potassium bromide infrared die, model 186-0025, made in Newark, CT. The powders were individually compressed using an Instron Corp. material testing instrument, model 2TDM, made in Canton, MA. The press's cross heads move at various, constant, pre-selected rates and the small compressive movement of 0.254 mm per minute was selected to insure some degree of pressure equilibrium. A total load of 3,000 kg cm^{-2} or 40,000 psi was applied slowly. At maximum pressure the direction of the cross heads was reversed, at the same rate,

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to obtain a decompression curve. In some cases the process was repeated twice to define the permanent effect of the first compression.

One sample of potassium nitrate was made damp by adding four percent colored water and grinding to a constant hue. The experiments were performed by Dominic Di Berardo of the PMB Branch of BRL.

D. Mercury Intrusion Porosity

Internal volume and pore size distribution of either whole or cleaved grains of black powder were measured by Jean Owens of Micromeritics Corp. located in Norcross, GA. The technique subjected samples to mercury pressures to 3.5×10^3 kg cm^{-2} or 50,000 psi forcing liquid into pores having diameters greater than 30A. The grains of black powder were degassed by a 100°C flowing nitrogen stream for 40 minutes. The technique has been described by Orr[27] and Adamson.[28]

Mercury, due to its high surface tension, does not wet most surfaces and pressure is required to force the fluid into small pores. Hence, the pore size is directly related to applied pressure and the relationship is given by equation 1.

$$Pr = -2 \sigma \cos \Theta \tag{1}$$

where P is pressure, r is the radius, σ is the surface tension of mercury (taken as 474 dynes cm^{-1}) and Θ is the contact angle (taken as 130° for most substances).

The distribution of such openings is given by a distribution function, $D(r)$,

$$D(r) = \frac{P}{r} \frac{d(V_T - V)}{dP} \tag{2}$$

where V_T is the total penetration volume and V is the penetration volume with openings greater than r.

[27] Clyde Orr Jr., *Powder Technol.*, 3, 117 (1969/1970).

[28] Arthur W. Adamson, *Physical Chemistry of Surfaces*, 2nd Ed., Interscience, NY, pp. 546-549, (1967).

E. B.E.T. Surface Area Measurements

In addition to the mercury intrusion measurements, Micromeritics Corp. also determined surface area by a single point B.E.T. technique using degassed whole grain samples and argon gas. This technique has been described in several texts, for example Flood,[29] where surface area is related to the number of argon atoms required to form a liquid film on a given surface. These measurements were extended to other samples by Eleonore Kayser of the Naval Surface Weapons Laboratory, NSW, in Silver Springs, MD, using the same type of Micromeritics equipment, model 2205.

F. Density Measurements

Even though density is defined clearly as gm/cm^3, the measurement of volume for powders and compressed masses can be ambiguous. For this reason some experimental approaches will be discussed.

1. "Tap Density" is a term that has been used to describe the weight of a powder or objects poured into a known geometry and allowed to settle by one or more gentle means such at "tapping". Such values are not used in this study.

2. "Bulk Density" depicts the volume element inscribed by its outer surface. The volume is measured by sinking a powder or object into mercury at atmospheric pressure. Such values do not include pore volume and are given in Table 1 as reported by Fitler.

3. "True Density" is obtained by measuring the volume of an object as equal to the amount of non-absorbed gases it displaces. In such measurements helium is usually introduced into an evacuated sample and fills small pores. Similar values are obtained by using ether as the displacement fluid for evacuated samples,[30] or using mercury displacement at very high pressures of mercury. In this study an ether displacement measurement was made on non-evacuated samples to estimate the samples' true density. Flow of ether into the sample was aided by placing the pycnometer filled with black powder and ether in an ultrasonic bath.

III. RESULTS

A. Black Powder and Charcoal S.E.M Microphotographs

Four black powder samples were chosen from the deviant lots for examination. Lots 1 and 6 are samples having slow flame spread rates of

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[29] E. Alison Flood, Ed., The Solid-Gas Interface, Vol. 1, Marcel Dekker Inc., NY, (1967).

[30] R. M. McIntosh and A. P. Stuart, Can. J. Res. B24, 124, (1946).

0.32 and 0.39 m/s as contrasted to lots 10 and 11 which have faster flame spread rates of 0.51 and 0.61 m/s. Further, lots 1, 6, and 10 are jet-milled oak products whereas lot 11 is a standard wheel-milled material of GOEX, Inc. The S.E.M. microphotographs are given in Figures 2. 3, 4, and 5. Stereo views were made but add little to the detail already shown and therefore are not presented. Potassium and sulfur x-ray maps were obtained but the concentration profiles did not show sharp boundaries as prominent features. The problem is the average particle size is about 15 microns or less and the x-ray maps depicted an average concentration to a depth of approximately 30 microns. Therefore such analysis represented an average concentration throughout the slab. For this reason those views have not been included.

The microprobe experiment was abandoned for black powder surface analysis, for although the sulfur and potassium x-rays were recorded simultaneously, and gave strong signals, the electron beam created a reaction path and the data could not be relied upon to depict the original sample. However, the microprobe did analyze the ash content of both extracted charcoals and calcium was found to be the dominant species and traces of silicon and phosphorous were noted. No other metals were detected. The ash content of maple charcoal was 2.3 percent and 11.9 percent was found for oak.

The S.E.M. microphotographs of the raw materials, oak and maple charcoals, are given in Figures 6 and 7. Extracted charcoals from black powder are shown for lot 8 in Figure 8 and for lot 11 in Figure 9.

B. Compression of Sulfur, Potassium Nitrate, and Charcoal

In the manufacture of black powder, damp meal typically containing materials of less than 25 microns, is pressed into a cake where various fabricators have used different compaction pressures. In general, a pressure is selected which will result in a finished grain density of approximately 1.7 and GOEX, Inc. employs a pressure no smaller than 246 kg cm² or 3,500 psi. All of the S.E.M. microphotographs, Figures 2-5, show considerable plastic flow and fusing among particles forming extensive agglomeration larger than the original particle size. Therefore a compaction experiment was performed to determine the relationship between flow and applied pressure.

The results of compressing pure ground sulfur, potassium nitrate and charcoal are given in Figure 10. The relationships shown can be linearized by presenting the logarithm of applied pressure as a function of compaction. The density of this sulfur cake was 2.00 gm/cm³ and that of the damp and dry potassium nitrate cakes were both 2.08 gm/cm³. Literature values[55] are 2.07 and 2.109 gm/cm³ for these compounds and thus, at the maximum pressure used, no significant voids exist in either material. In these cases the decompression curve is almost vertical showing little elasticity and recompression forms a small envelope about the decompression curve. The oak and maple charcoal compression curves

50 microns
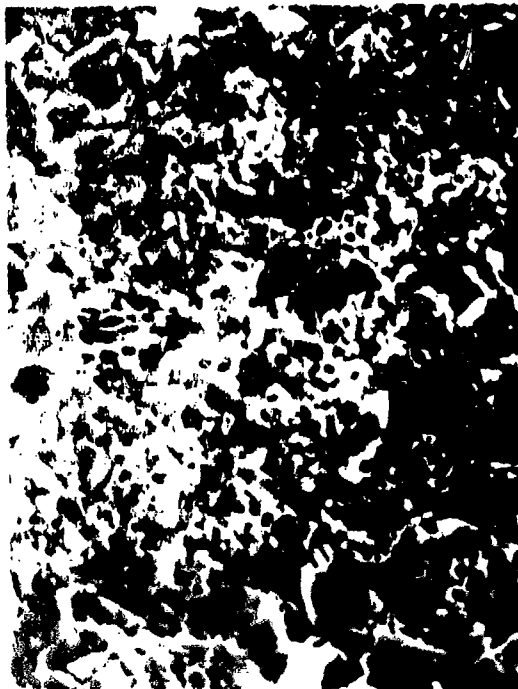
100 microns

Figure 2. Black Powder, Lot 1

50 microns
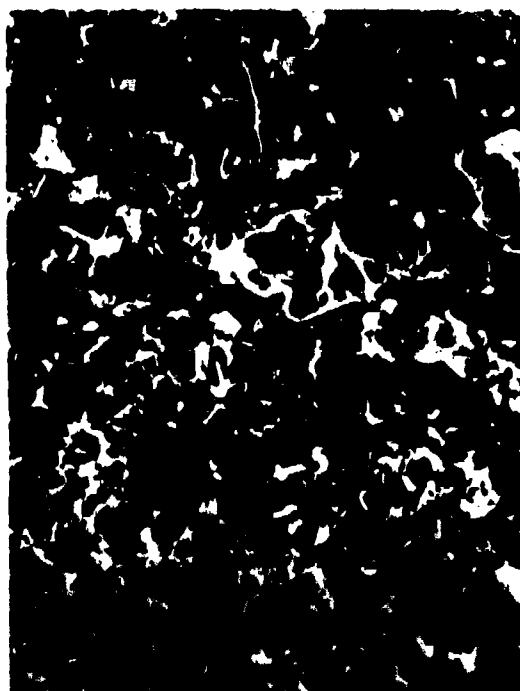
100 microns
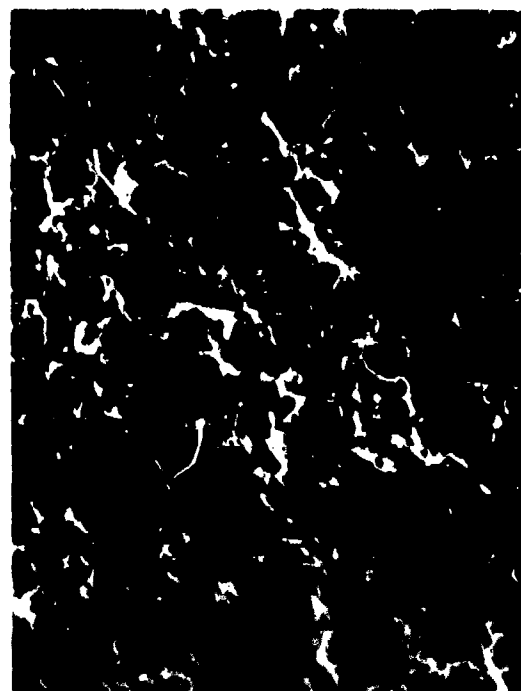
Figure 3. Black Powder, Lot 6

20
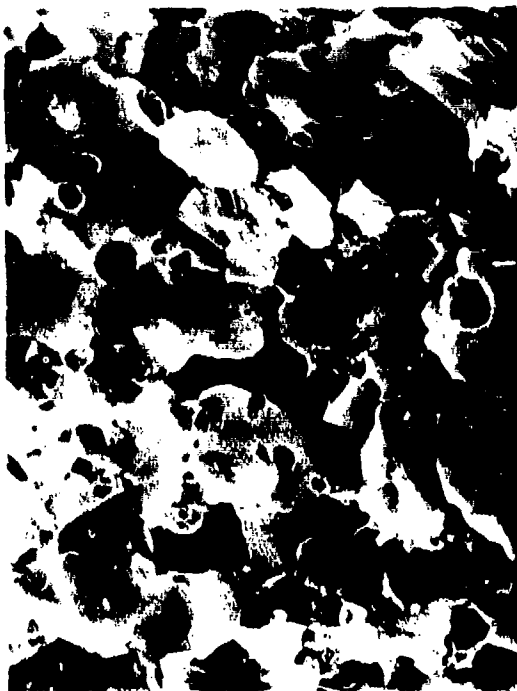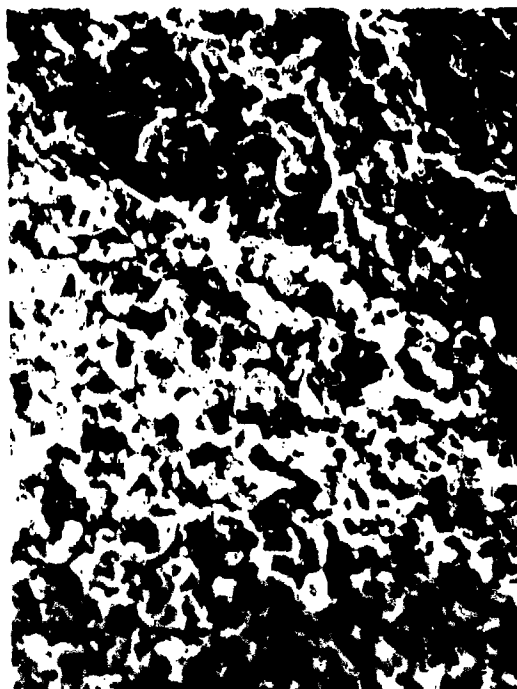
50 microns
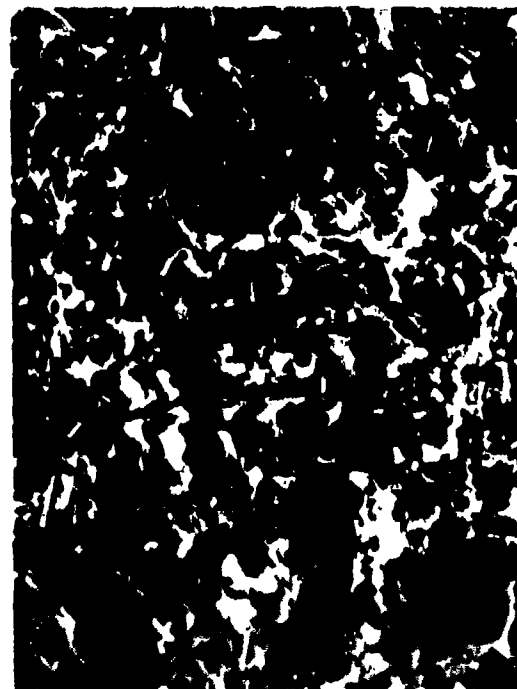
100 microns

Figure 4. Black Powder, Lot 10
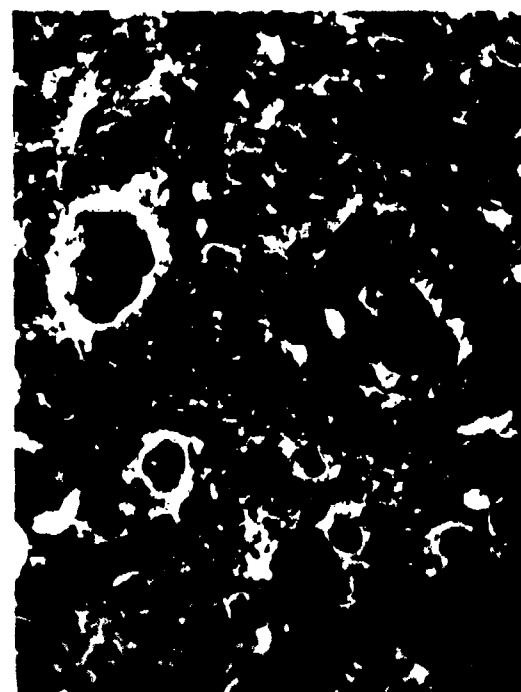
Figure 5. Black Powder, Lot 11

22

OAK

100 microns

OAK

100 microns

MAPLE

100 microns
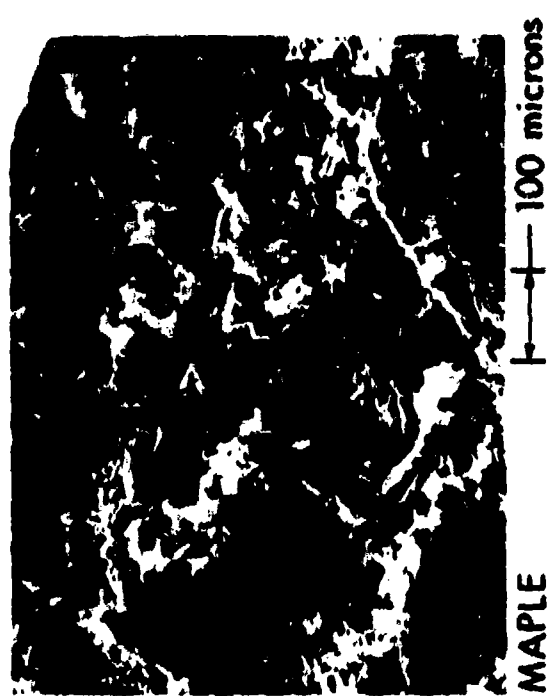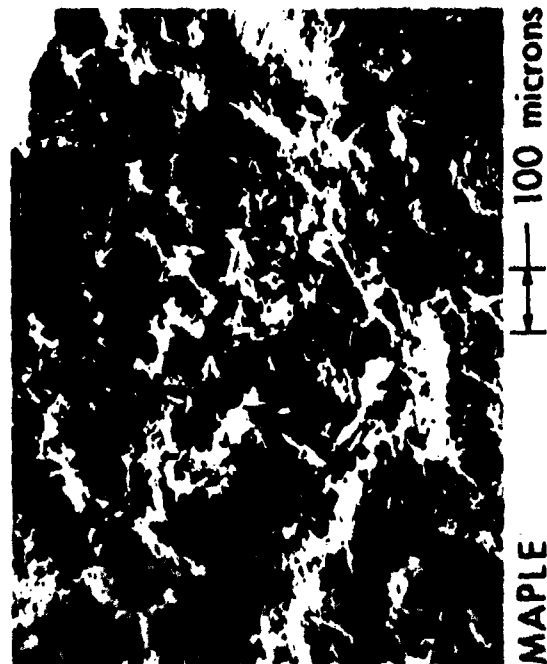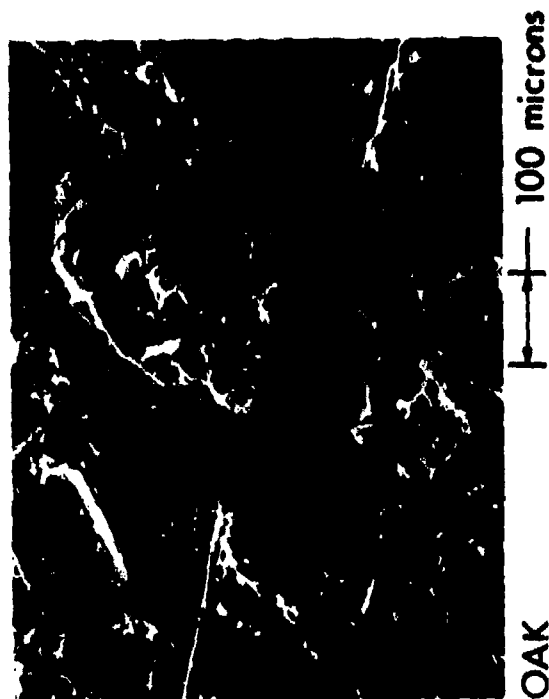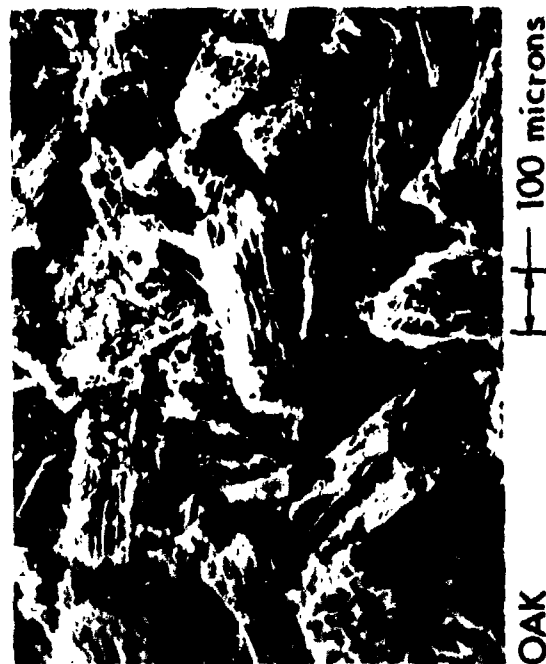
MAPLE

100 microns

Figure 6. Oak and Maple Charcoals

23
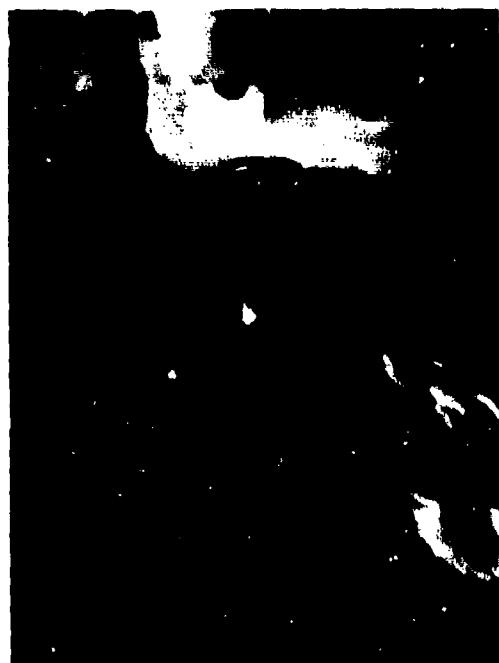
Figure 7. Oak and Maple Charcoals

30 microns

10 microns

100 microns

20 microns
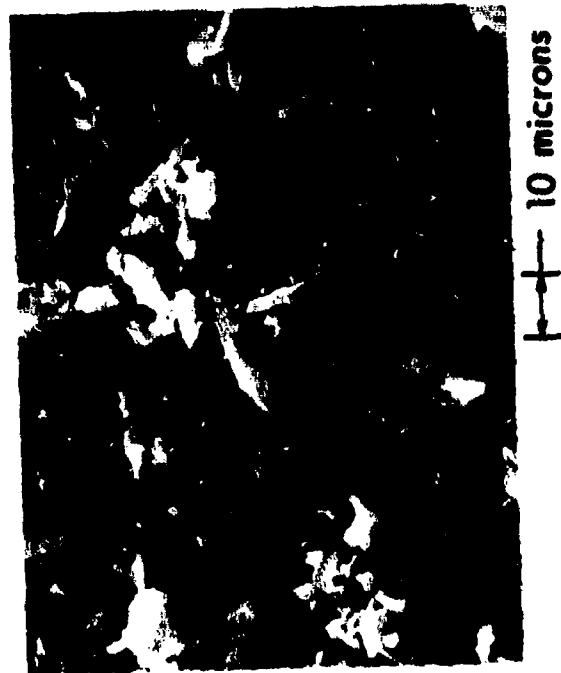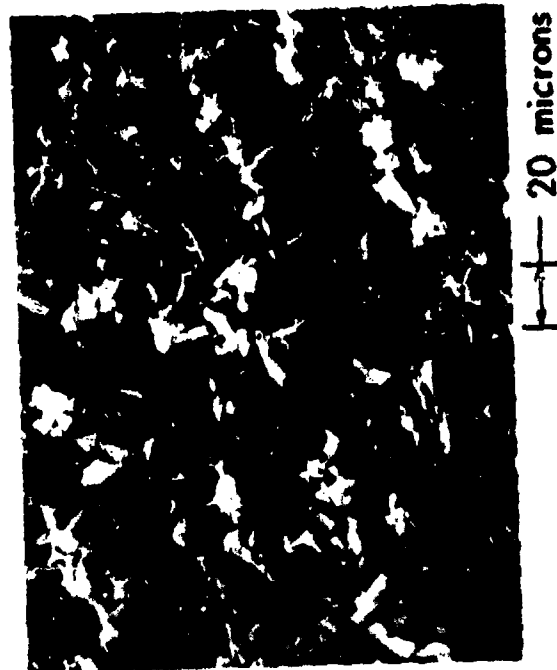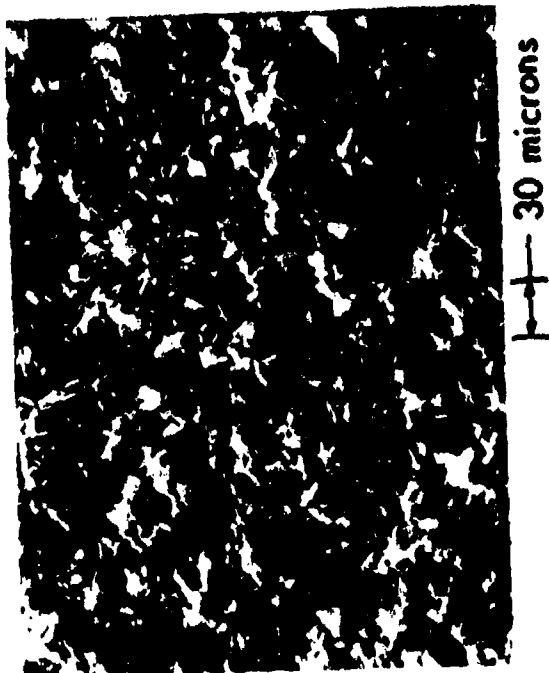
Figure 8. Oak Charcoal Extracted from Lot 1
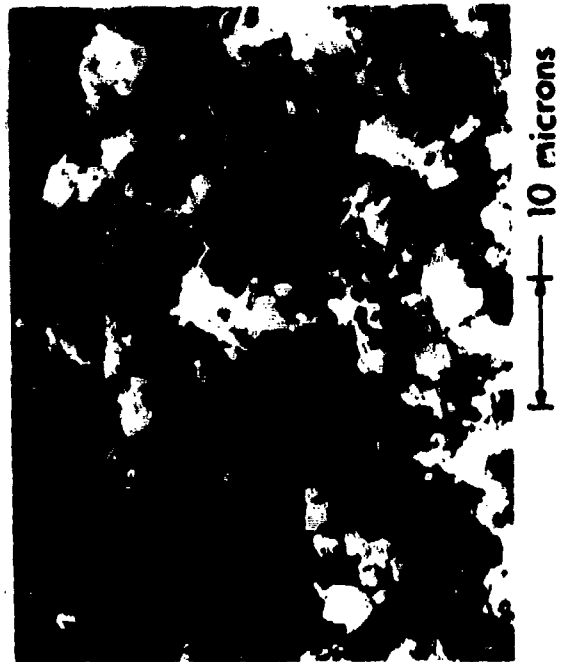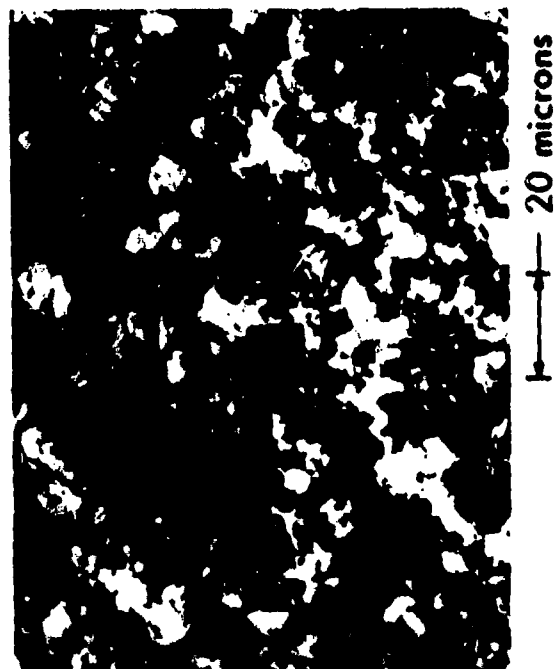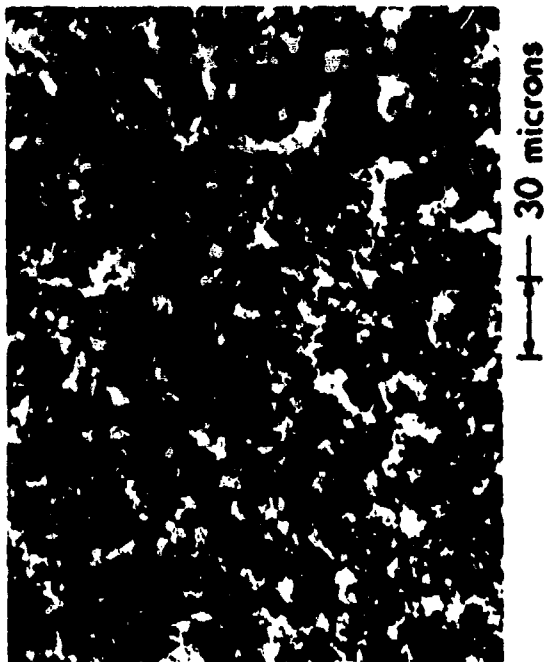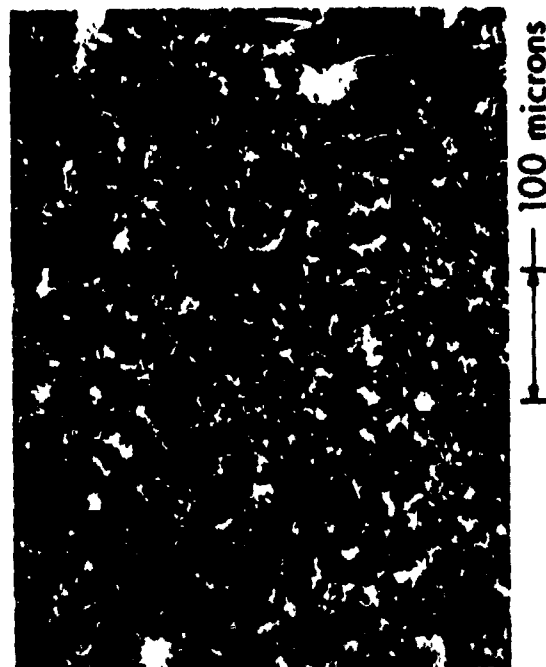
Figure 9. Maple Charcoal Extracted from Lot 11
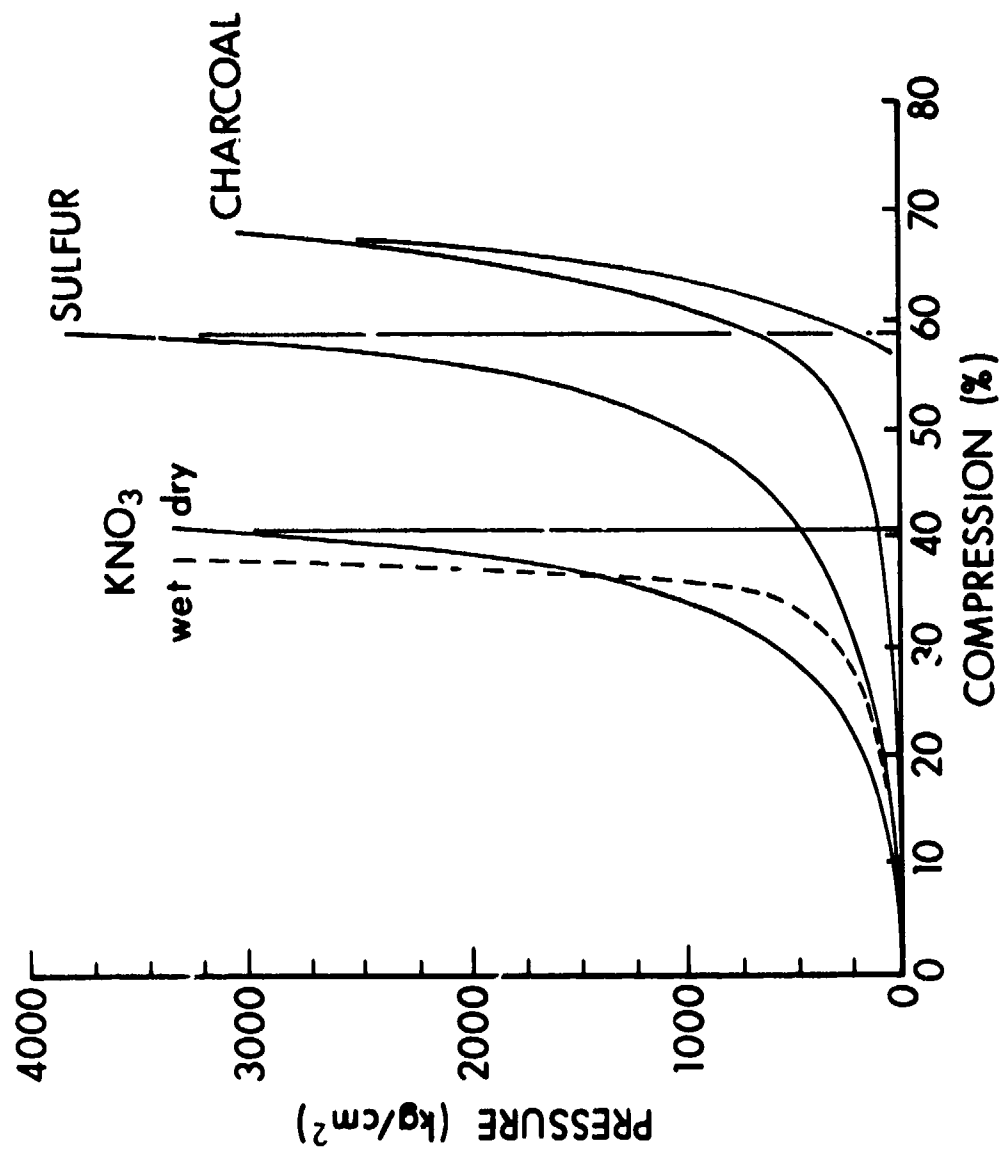
Figure 10. Effect of pressure on potassium nitrate, sulfur, and charcoal

were identical, but a compressed density of 1.01 gm/cm^3 was measured for the maple charcoal and a density of 1.16 gm/cm^3 was measured for oak to an accuracy of 0.5 percent. Values were obtained from the sample weight and die dimensions at maxiumum pressure. The decompression curves for both samples showed considerable elasticity. Interestingly, each sample was removed as a powder from the die and obviously the organics did not act as a binder.

In an attempt to interpret the compression density values, the densities of both charcoals were measured by an ether displacement technique using a pycnometer. The density values were 0.99 and 1.17 gm/cm^3 and were accurate to 1.0 percent. They were equal to those measured in the compression experiments. After compression, the charcoal originally passing through an 80 but not a 100 mesh screen, was again sieved and the results are given in Table 2. The values show that many pieces of charcoal were broken by the large applied force.

TABLE 2. CHARCOAL SIEVED AFTER COMPRESSION

| Screen Size Mesh Units	Oak* %	Maple* %
100	24	51
120	12	8
200	38	26
340	26	15

*% by weight

C. Mercury Intrusion Porosimetry

Three different samples of 1-2 grains of whole grain black powder, lots 6 and 10, were evaluated to determine the reproducibility of both the sampling and measuring techniques. Data are given in Figure 11 and 12 showing the cumulative amount of mercury forced into pores of different radii by various pressures. The d. *a have been normalized at 200 psi or 14 kg/cm^2 and shows a good degree of reproducibility. One sample each of lots 1 and 11 was evaluated and this data is given together with a representative penetration curve for lots 1 and 6 in Figure 13. These same four lots were also evaluated by cutting the grains in half. These data are given in Figure 14. In all cases the cut or whole grains were intact after pressure was released.

D. B.E.T. Surface Area Measurements

The surface area of whole grains was measured by a B.E.T. analysis. All of the deviant lots were evaluated as well as GOEX, Inc. and CIL
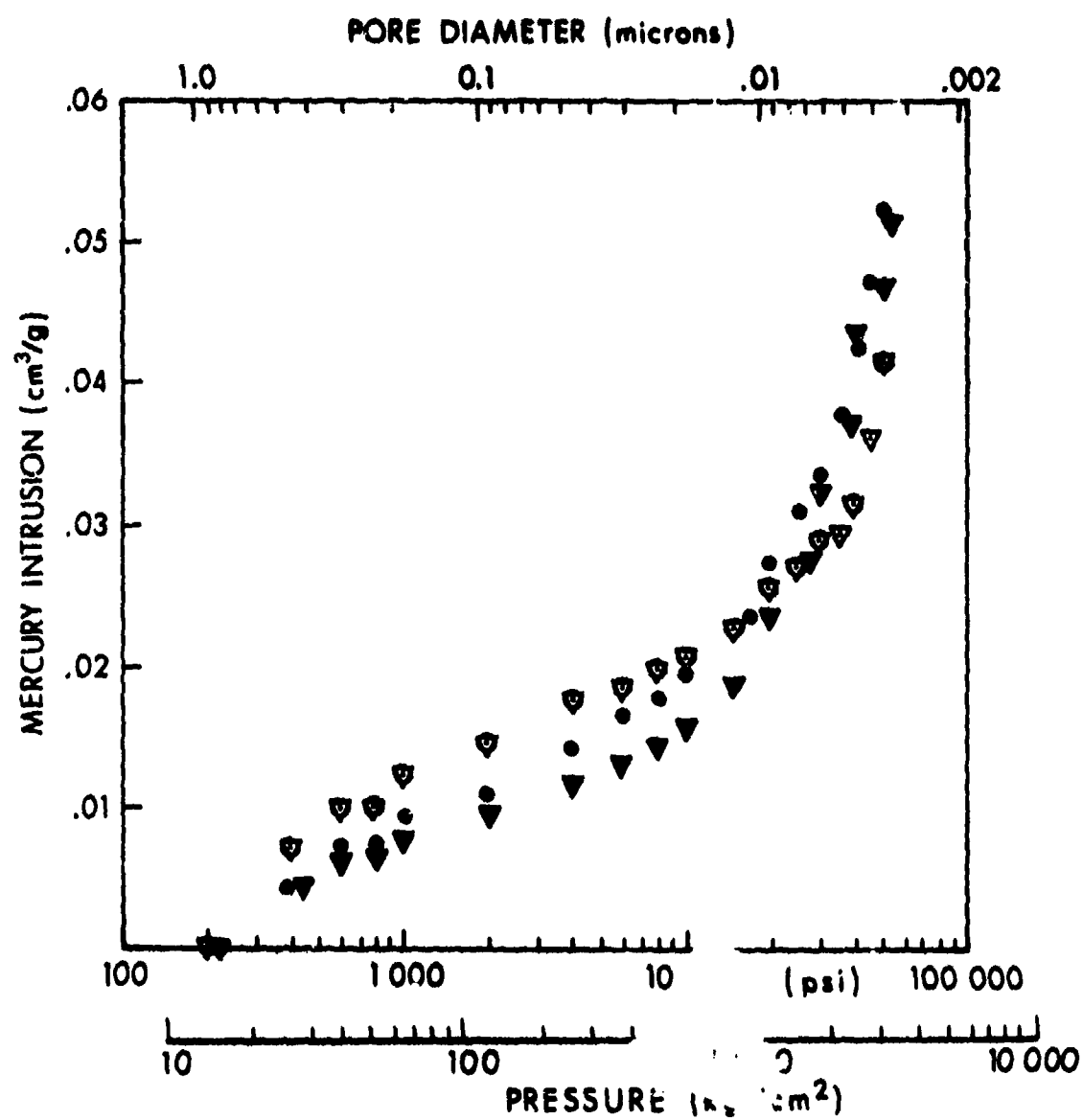
Figure 11. Volume of mercury entering three samples of black powder - Lot 6
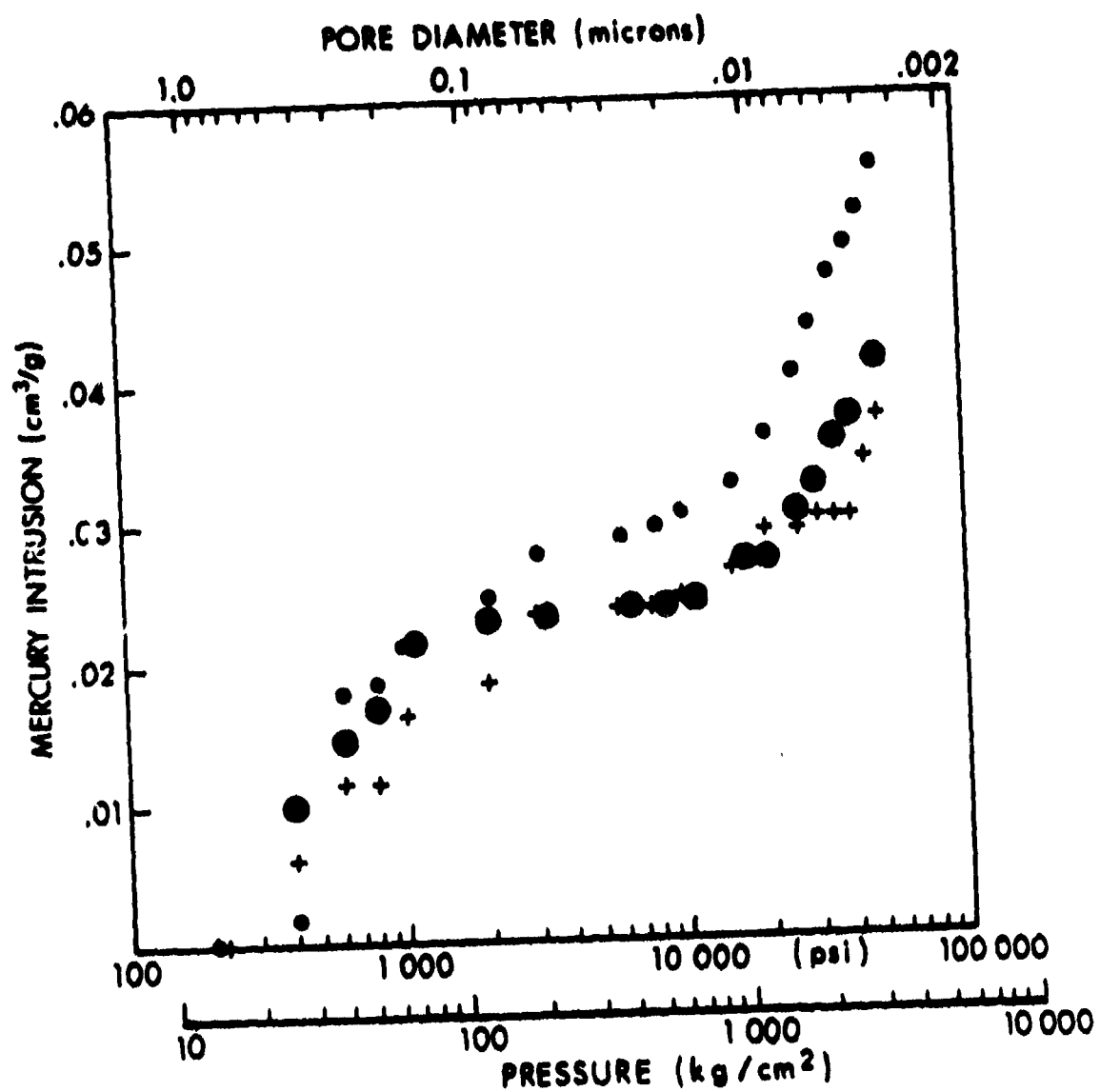
Figure 12. Volume of mercury entering three samples of black powder – Lot 10
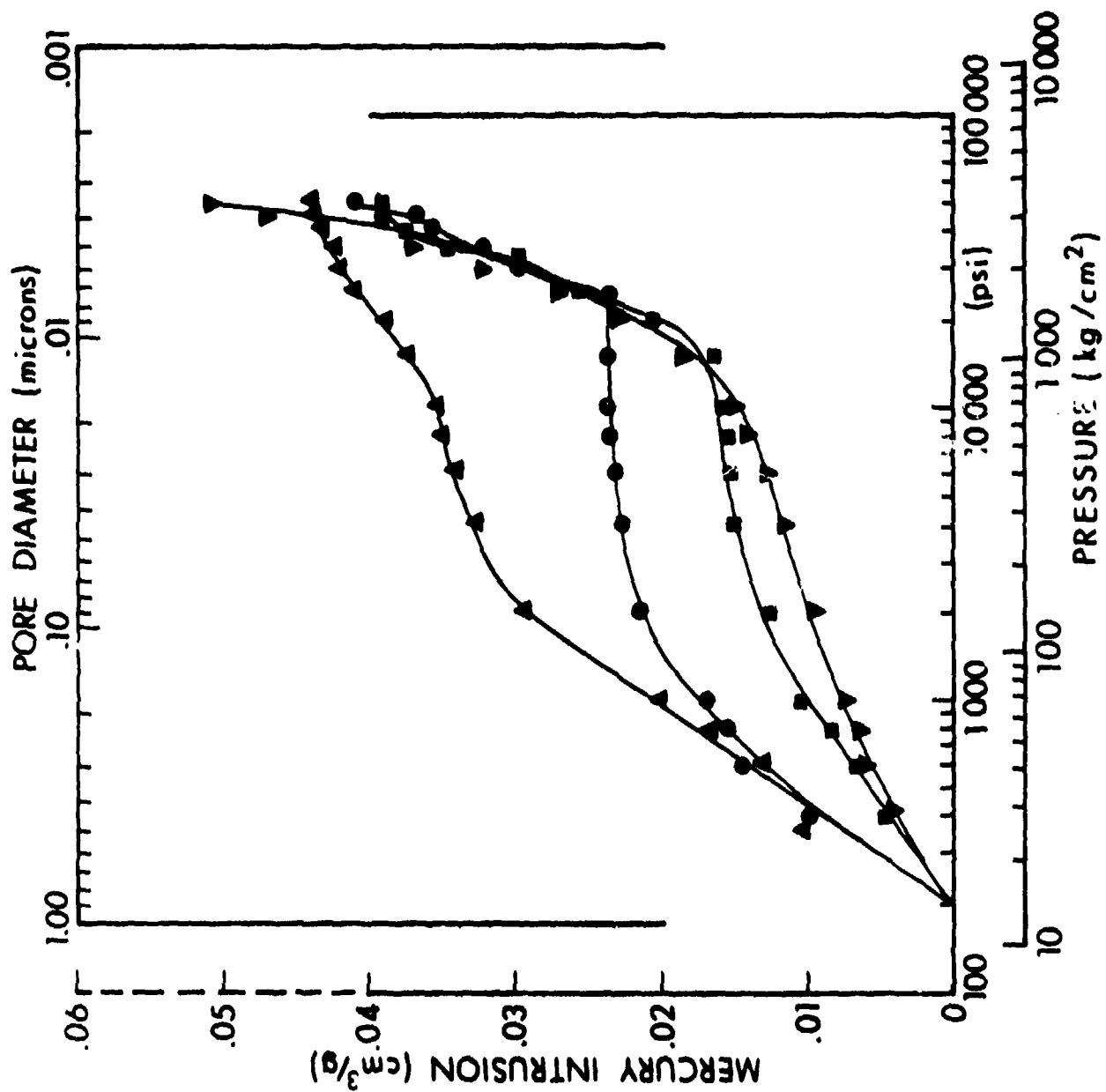
Figure 13. Volume of mercury entering whole grains of black powder: ▲ -lot 11, burn rate 0.60 m/sec; ● -lot 10, burn rate 0.51 m/sec; ■ -lot 1, burn rate 0.32 m/sec; and ▼ -lot 6, burn rate 0.35 m/sec.

51
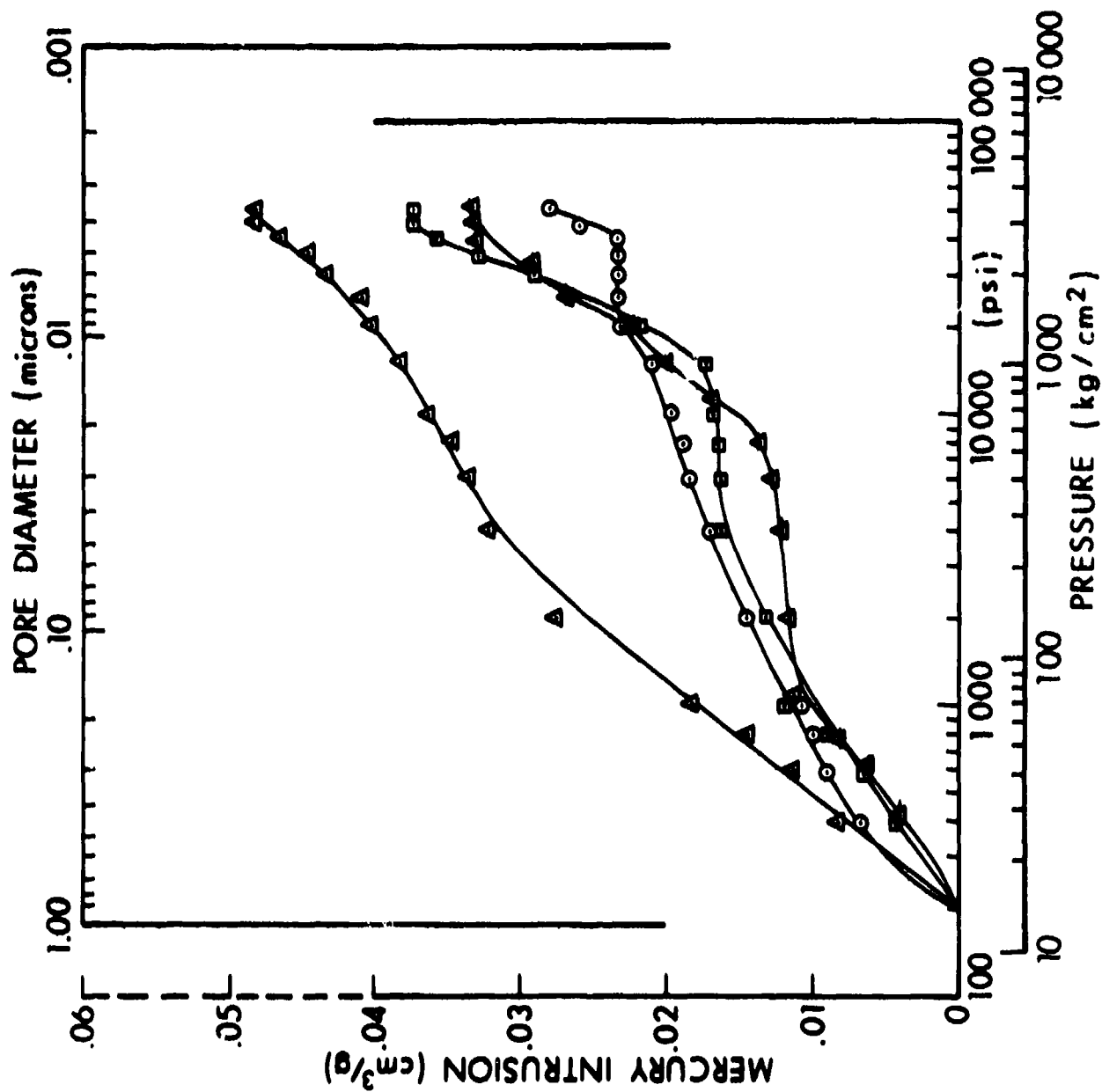
Figure 14. Volume of mercury entering half grains of black powder: ▲ –lot 11, burn rate 0.60 m/sec; O –lot 10, burn rate 0.51 m/sec; □ –lot 1, burn rate 0.32 m/sec; and ▼ –lot 6, burn rate 0.38 m/sec.

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black powder. One lot, lot 6, was examined in detail to determine the variance and precision associated with sampling and measurement. The data are given in Table 3 and surface areas ranged from 0.5 to 1.0 m^2 gm^{-1}. Each individual sample was evaluated at least twice.

E. Density Measurements

The density of whole grains of black powder was measured by an ether displacement technique and values are given in Table 3. They were measured to an accuracy of one tenth of a percent.

The ether displacement density of charcoal extracted from lot 11 was 1.43. The densities of the maple and oak charcoals supplied from the Hardwood Charcoal Co., were 1.01 and 1.16. Reproducibility was as noted above.

IV. DISCUSSION

A. Black Powder and Charcoal S.E.M. Measurements

The microphotographs of deviant lots 1, 6, and 10, made by the jet mill process, look much alike. They are shown in Figures 2-4. Each figure shows two different views of a particular grain and these views are shown at two different magnifications. The view at 300X was attempted to get a general representation. A smaller subsection is shown at 1000X to provide greater detail. Higher magnifications were recorded but they lose the general nature of the features shown. Sulfur and potassium nitrate cannot be individually discerned; however from the relative abundance most of the features represent potassium nitrate.

Figure 2 shows a large isolated charcoal particle; clearly, compression did not fill its pores nor did it crush the charcoal. Thus in this instance incorporation of material into the charcoal pores, a process that has been ascribed to the wheel mill process,[5] did not take place. In all figures considerable plastic flow and particle deformation are shown and many consolidated regions exist that are much larger than the original particle size of 10-15 microns. Figure 4, deviant lot 10, shows particles which appear more intact and are individually more discernable. Also the degree of plastic flow seems less. This leads to a greater degree of openness between particles.

The S.E.M. photograph of the GOEX, Inc. ball milled material, lot 11, Figure 5, is different from the three-jet milled samples. Large clumps of material are observed as well as large void regions. Again one piece of charcoal is evident and its pores are not filled, nor is it crushed. The pores are 2.8 ± 0.9 microns wide. In general, material seems to have flowed around the charcoal and the photograph shows almost geological folding. Stress lines and spherical pockets are noted in

TABLE 3.   SURFACE AREA OF BLACK POWDER AND ETHER DISPLACEMENT DENSITY

Lot Number	Surface Area* m^2 gm^{-1}	Surface Area** m^2 gm^{-1}	Density gm cm^{-3}
1	0.50 0.53	0.588 0.588	1.91
2	0.73 0.80		1.87
3	0.54 0.58		1.88
4	0.060 0.060		1.89
5	0.58 0.58		1.85
6***	0.56 0.55 0.52 0.57 0.53 0.51 0.62 0.52	0.588 0.587	1.86
7	0.55 0.58		1.85
8	0.52 0.52		1.83
9	0.67 0.63		1.82
10	0.66 0.62	0.639 0.630	1.85
GOEX - Lot 11	0.66 0.82 0.76	0.801 0.796	2.02
GOEX 75-44	0.85 0.85 0.82		
GOEX 75-61	0.71 0.73		
GOEX 75-2	0.71 0.69		
CIL 7-11	0.58 0.57		

*
 NSW
**Micromertics Corp.*
***Average is 0.55 ± 0.04.*

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consolidated regions. The grain is not as homogeneous in appearance
as the jet-milled grain.

In summarizing the S.E.M. microphotographs, compaction seems to form
a fused conglomerate mixture laced with a maze of interconnecting passage-
ways.

The S.E.M. microphotographs of oak and maple charcoals are given in
Figures 6 and 7. Oak charcoal appears as separate lumps having a clearly
defined cell structure. The maple sample looks torn apart, perhaps by
the grinding action of the mortar and pestle. However, both samples
show extensive cell structure and broken and chipped pieces dominate.

One series of S.E.M. microphotographs of oak, Figure 8, which was
ground in the jet mill and extracted from lot 1 black powder, Figure 9,
looks similar to maple charcoal obtained in the same manner from lot 11
ground in a wheel mill. The particles seem to be of similar size and the
only difference is the maple appears more rounded. No large charcoal
pieces are evident in the maple photograph as was seen in its black powder
counterpart but the view may not be totally representative.

In summary, it appears that grinding by either method produces a
fine powder which has a multiplicity of edges, broken pieces and con-
voluted surfaces. The grinding process breaks down the gross structure
of charcoal, forming pieces and fragments such that the two charcoals
look much alike.

B. Compression of Sulfur, Potassium Nitrate and Charcoal

Noting the extensive flow in grains it became desirable to quantify
the effect of pressure on black powder meal. Damp meal, that is generally
4 percent in moisture, has been pressed into a cake using various compac-
tion pressures. Values of 1900 psi[5] (134 kg/cm^2) to at least 3,500 psi[8]
(246 kg/cm^2)[8] has been employed and some work was done at 8,000 psi[31]
(563 kg/cm^2). The range of these values raised the questions, "do these
compaction pressures span the individual yield points of the ingredients
and is a particular pressure threshold required to induce plastic flow?"
To answer these questions an experiment was performed to determine if
the change in volume, or plastic flow, was uniform as the applied pres-
sure was increased.

The effect of pressure on the pure materials was determined and
shown in Figure 10. From the curvature, it can be seen that each
material flows throughout the applied pressure range. This is a
reminder that the applied pressure is a global value but the microscopic
pressure exerted between particles can be much greater. One compression

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[31]*Memorial Des Poudres Et Salpetres*, Vol. 6, Chapter II by M. Vieille,
Gauthier-Vallars Et Fils, Imprimeurs-Libraires, Paris, pp. 256-391, (1893).

curve using damp potassium nitrate, four percent moisture, lies well under its dry counterpart showing that water reduces friction, allowing particles to slide. Thus, applied pressure and water content are coupled phenomenon which govern grain density.

It was noted that both potassium nitrate and sulfur formed smooth glass like surfaces at the die faces. Such a surface could seal the pellet and this effect could explain why black powder pelletized in a pellet press exhibits slow relative quickness values.[5] Additional descriptions of this effect can be found in the Ordnance Explosive Train Handbook[32] that describes the burning of a train of several pellets as a succession of puffs caused by burning through the "glazed interface". In such applications, these effects are reduced by pressing pellets as interlocking cones to minimize the effects of burning through a sharp boundary.

In addition to the surface effect of pressed pellets, there is a density profile that should be considered when pressing to a given grain density.

The charcoal compression curve is similar to that of potassium nitrate and sulfur. From the facts that bulk density at maximum compression and ether displacement density are equal and that compression fractures charcoal (Table 2), it is clear that pressure fractures some of the charcoal into smaller fragments which then bend elastically to fill the die leaving very little space between particles. With a density of 0.99 for maple and 1.17 for oak charcoal, both far removed from the amorphous carbon value[33] of 1.8 - 2.1, it is concluded that the density of charcoal reflects an open structure.

The compression results show a gradual change in volume with applied pressure. This reflects that applied pressure is a global value and the associated microscopic pressure between small areas of two touching particles is much greater. The smooth exponential curves show no sharp transitions that could be associated with yield points nor are any threshold values indicated.

The effect of compression on black powder meal is yet to be done but the effect of pressure is to increase density. Vieille[31] in 1893 demonstrated the relationship between black powder bulk density and burn rate. The data was reproduced by Urbanski[1] and his graph is given in Figure 15. Obviously the data relate to a process and materials different from those used today, but the trends should still persist. This same type of

[32] *Naval Ordnance Laboratory, Ordnance Explosive Train Designers Handbook, NOLR 1111, April 1952, Naval Ordnance Laboratory, White Oak, MD.*

[33] *Robert C. West, ed., Handbook of Chemistry and Physics, 51st Ed., The Chemical Rubber Co., Cleveland, Ohio (1970-1971).*
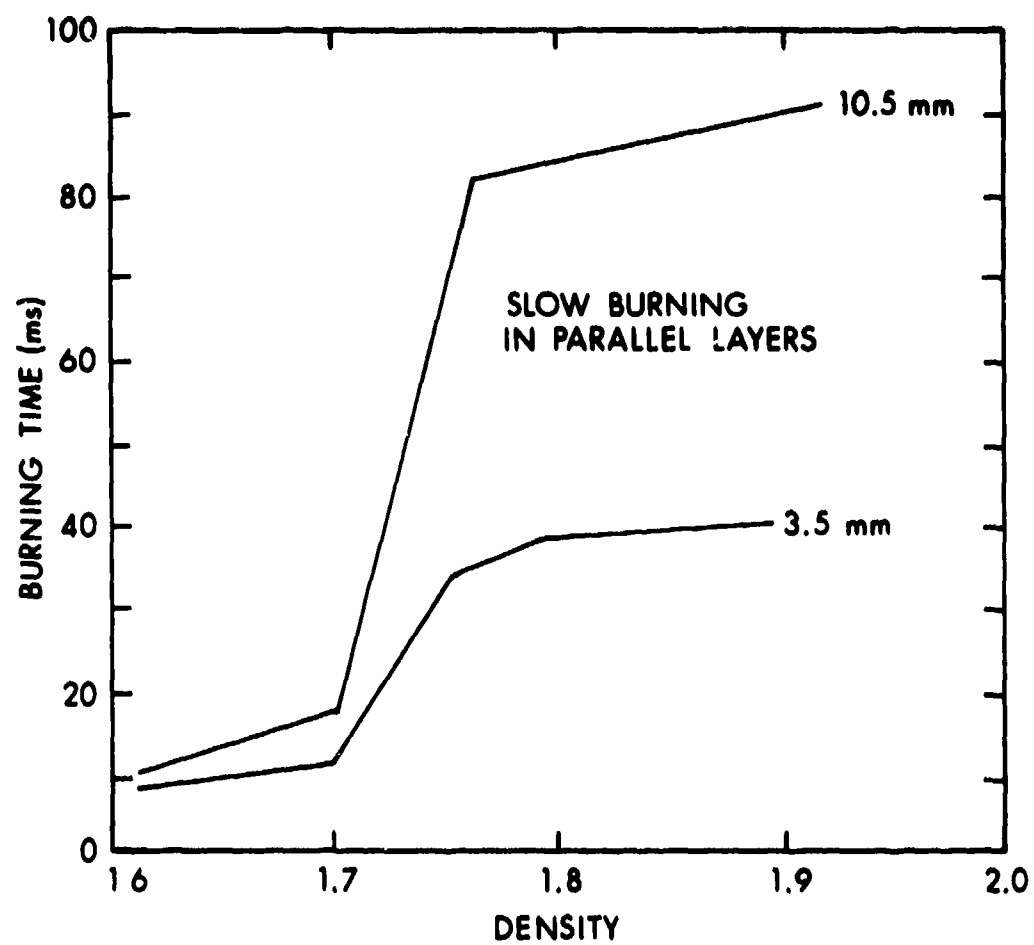
Figure 15. Relationship between burning rate and density of black powder reproduced from reference 1. Dimensions refer to pellet size.

relationship was shown to be operative in pyrotechnic mixtures where
burning rate was given as a function of compaction pressure.[34]

Vieille investigated the large density neighborhood of 1.44 to 1.85.
In contrast, present experiments, using material that was available,
define a more restrictive neighborhood and the parameter is not illus-
trated as clearly. Since Vieille's reference is not readily available,
the liberty has been taken to translate his summary. This translation
was undertaken by Eli Freedman of our laboratory (BRL-Applied Ballistics
Branch) and is given as an appendix.

The flame spread rate and densities for the deviant lots, given in
Table 1 are shown as a scattered presentation in Figure 16. A functional
relationship appears to exist but its exact nature is not given by these
limited data. The scatter leads to the belief that compression condi-
tions were not identical; however, it is clear that flame spread rate
decreases as density increases.

C. Mercury Intrusion Porosimetry

Equation (1) relating pore size and applied pressure presumes that
the pores are a collection of right cylinders of different radii. In
practice small openings exist leading to a larger volume element. Such
a situation has been termed an "ink bottle effect" and when encountered
will lead to an overestimate of the number of pores that exist at a
particular radius. For loose powders, corrections are attempted by
measuring the mercury extruded as a sample is depressurized. In accor-
dance with equation (1) the neck of the "ink bottle" will empty as well
as the "bottle" itself but at different pressures. For compacted
material, such a black powder equilibrium may be too slow to attempt this
correction depressurizing technique. These problems should be considered
when examining the intrusion data and the indicated radii should be con-
sidered as approximate values. However, the mercury volume entering a
grain is exact.

In examining the S.E.M. microphotographs of black powder many voids
are apparent with radii of 10 microns or less. For mercury to pene-
trate the grain and reach these voids it must intrude through passage-
ways that are small and are not particularly apparent in the view of the
plane presented. Therefore, in filling such a void through a small
passageway the mercury will fill the system indicating a pore volume
having the radius of the passageway. The mercury penetration data,
including this effect, are given in Figures 11, 12, 13, and 14. All of
the data have been normalized to 200 psi or 14 kg/cm^2. Three different

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[34]A. A. Shidlovskii, "Principles of Pyrotechnics", Air Force Systems
Command, Report No. AD-A001859, 23 Oct 1974, Wright Patterson AFB, OH,
Translation of Izd Masimostroyeniye, Oxnovy Pirotekhniki, 3rd Ed.,
Moscow (1964).

38
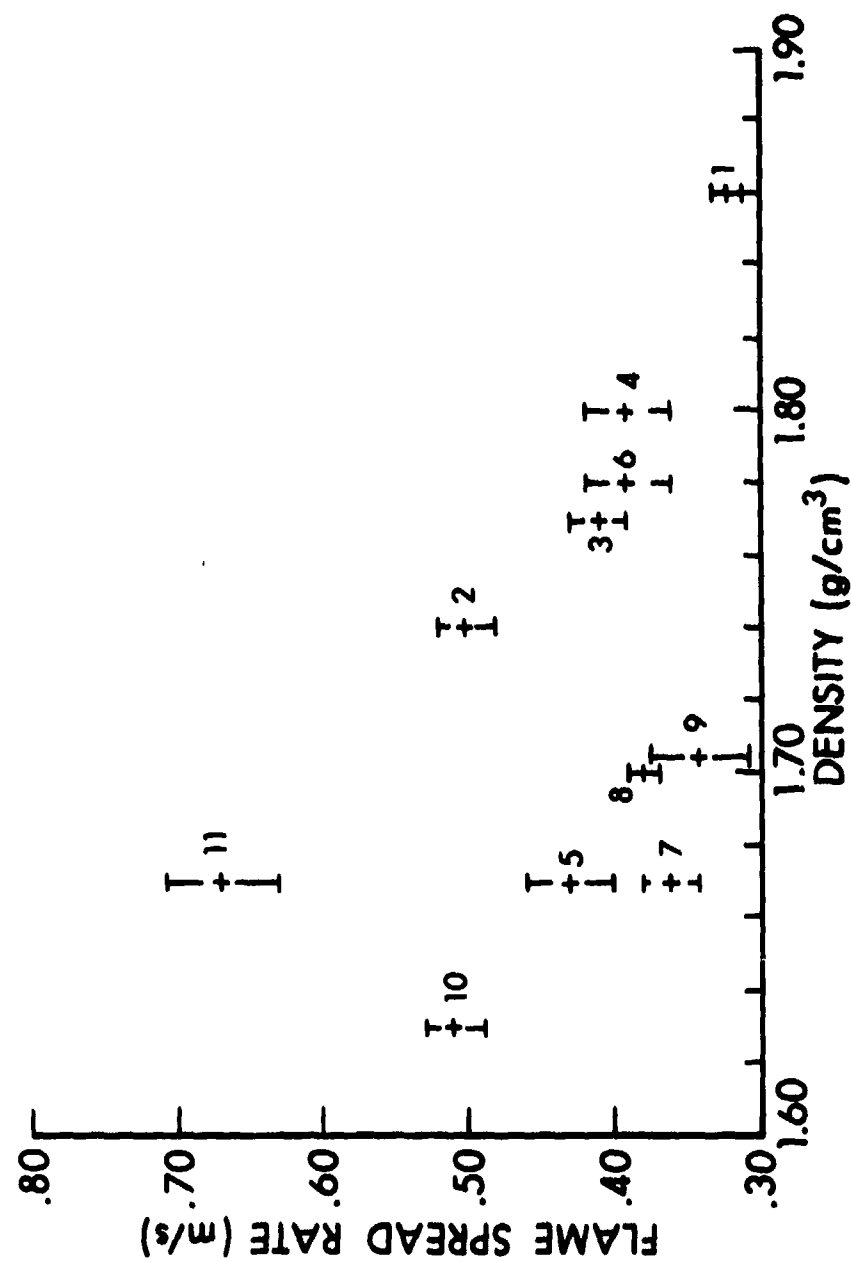
Figure 16. Relationship between flame spread rate and density. Subscripts refer to deviant lot number.

whole grain samples of lots 6 and 10 were evaluated, Figures 11 and 12, to determine the reproducibility of the sampling and measuring technique. The penetration data for these samples shows fair agreement and most of the mercury entered through passageways between 0.1 and 1.0 micron / meter. The experiment was expanded to include lots 1 and 11 and these data are presented in Figure 13 for whole grains. The experiment was repeated on new samples of these lots where the grains were first fractured in two. The penetration volume is presented for half grains in Figure 14. The data for lot 10 appears to be in error, and for this sample some of the grains may have been crushed by the applied pressure.

The mercury intrusion data for the four lots of black powder are nearly the same for either whole or cut grains; neither group showing major differences in penetration pattern. On this basis, it is concluded that the graphite coating does not seal the grain to pressurized mercury. The two experiments also show that mercury must be able to reach the interior of a grain since cut and whole grains host the same volume of penentration. If mercury intrusion had been related to the black powder surface, then penetration between whole and cut grains would have been approximately proportional to the ratio of the surface area of a sphere to two corresponding hemispheres or 2/3. This was not found to be so and the conclusion is that the mercury penetrated throughout the grain.

The distribution of passageways was calculated using equation (2) and the data of Figures 13 and 14. The calculation is primarily dependent on the slopes shown; therefore, it is not affected by the normalization. The distribution is given in Figures 17 and 18. A correlation exists between the number density of passageways between 0.2 and 0.1 micron and flame spread rate. No such relationship was evident for the smallest of radii and this range will be discussed later.

D. B.E.T. Surface Area Measurements

The intrusion data can be used to calculate internal surface area but because of the "ink bottle effect" the result would be suspect. For this reason the surface area was measured by the B.E.T. technique and the results are given in Table 3 and plotted as a function of burning rate in Figure 19. The standard deviation for four samples of lot 6 is 6.6 percent and similar agreement on replicates of the same sample was found. It is clear that an increase of internal surface area increases burning rate. The graph, Figure 19, contains several markedly different black powder samples including the jet-milled oak deviant lots, maple GOEX Inc., powder and one CIL sample. The latter two producers used the ball-wheel mill process. Overall, the samples appear to follow the same function even in the small neighborhood tested where the density of black powder does not vary to any great extent. It remains to the future to develop a broader neighborhood for investigation. Under these constraints the relationship between burning rate and the degree of openness of a grain of black powder seems reasonable.

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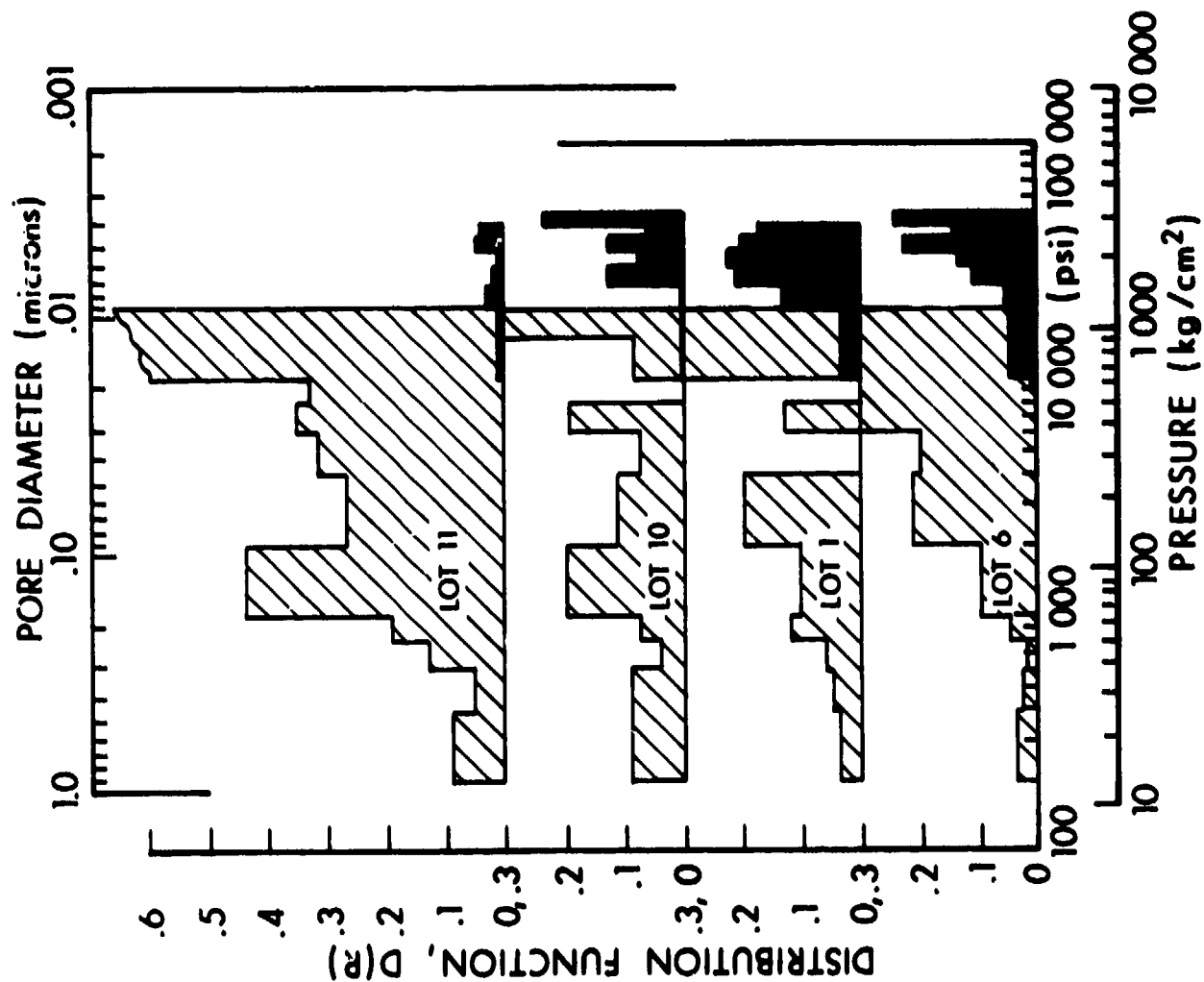
Figure 17. Passageway diameter distribution for whole grains of black powder. Dark portion repressed by a factor of 10.

41
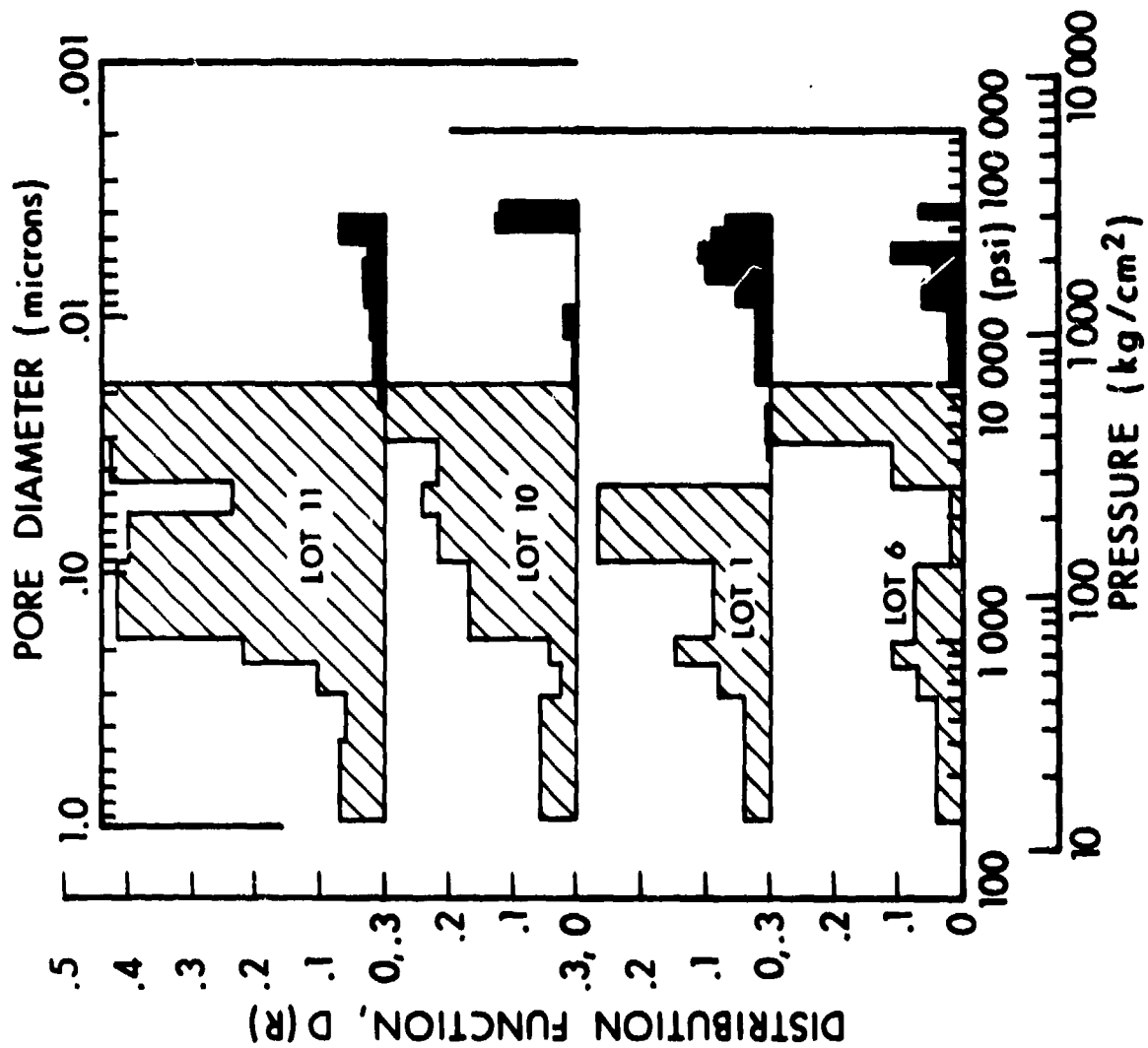
Figure 18. Passageway diameter distribution for half grains of black powder. Dark portions repressed by a factor of 10.

42
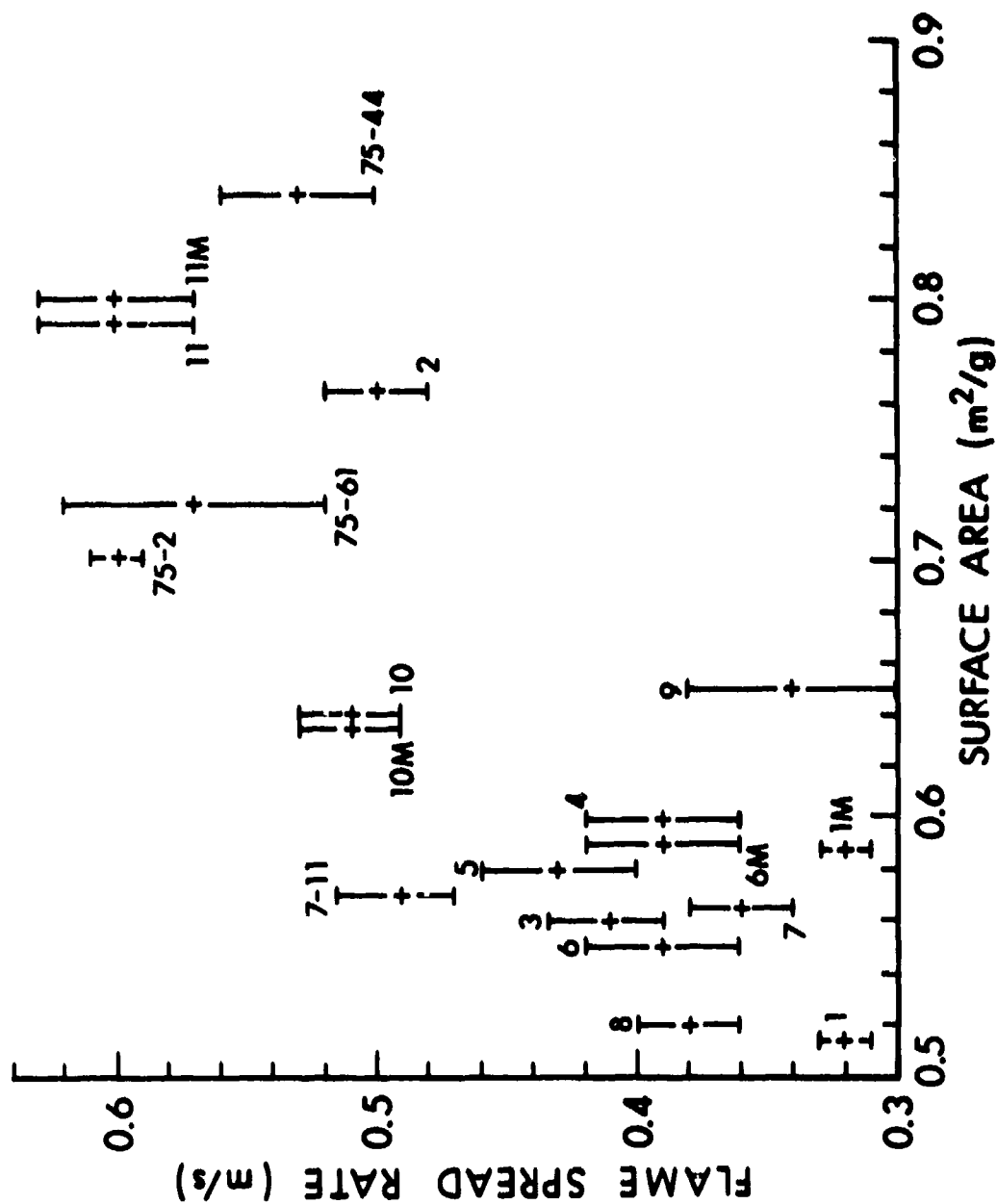
Figure 19. Relationship between flame spread rate and surface area. Subscripts refer to lot number: 1-10 deviant lots; 11, 75-2, 75-61, 75-44 are GOEX material; 7-11, CIL materials; and M subscript measured by Micromeritics Corp., others by NSW.

The surface area of charcoal[22] is about 1000 m^2 gm^{-1} and since charcoal consititues 15.6 percent of black powder, an upper value of 156 m^2 gm^{-1} would be expected. The measured values are much less, about 0.5 to 1.0 m^2 gm^{-1}. Therefore, it is concluded that almost all of the charcoal particles are sufficiently sealed to greatly reduce gas absorption. This could explain why burning rate was not found to be a function of the original surface area of charcoal or other carbon materials.[22]

It also should be pointed out that the surface area measurements were extended from the original four samples to include all of the deviant lots. This was performed as a matter of convenience rather than preference over the pore size measurements, for at the time of analysis it was more expedient to extend the surface area measurements.

E. Density Measurements

The total free volume can also be calculated from the bulk density of black powder, $(D_{B.P.})$, the density of the separate ingredients, (D_i), and percent composition. Such values were taken from Table 1 and the following relationship, equation 3, was applied to a 100 gm sample. Ash and moisture content were not included.

$$\frac{100}{D_{B.P.}} = 100 \left[\frac{\% \ KNO_3}{D_{KNO_3}} + \frac{\% \ S}{D_S} + \frac{\% \ C}{D_C} \right] + \text{free volume.} \tag{3}$$

Using a density of 1.17 for oak and 1.43 for maple charcoal (determined from charcoal extracted from lot 11) the total free volume was calculated. Values are given in Table 4 and Figure 20. They incorporate both isolated voids and accessible volume elements. Equation (3) can be used to show that the density of charcoal is a sensitive parameter that affects the free volume of black powder when a particular compression pressure is selected for manufacture. Moreover, compressing black powder to a particular density using different density charcoals will produce grains with varying degrees of free volume. One problem yet to be addressed is that no knowledge exists on the density variations for lot-to-lot procurement of charcoal. If a variance is found, compaction pressure and grain density will have to be adjusted accordingly to obtain the same free volume or a particular density.

In an attempt to measure the volume accessible from outside of the grain, the true density (D_T) was measured by an ether displacement technique. These values and the bulk density $(D_{B.P.})$ from Table 1, were related to the free volume for a 100 gm sample by the equation:

$$\frac{100}{D_{B.P.}} = \frac{100}{D_T} + \text{free volume.} \tag{4}$$

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TABLE 4. FREE VOLUME IN BLACK POWDER

Lot Number	Equation (5) cm^3/100 gm	Equation (4) cm^3/100 gm	Mercury Intrusion- to 0.1 µ cm^3/100 gm	Mercury Intrusion- to 0.003 µ cm^3/100 gm
1	2.22	1.41	1.3	3.9
2	3.61	3.99		
3	4.67	3.22		
4	1.64	2.65		
5	7.79	5.93		
6	3.93	2.72	1.0	5.1
7	6.36	5.92		
8	6.20	4.18		
9	7.23	3.88		
10	8.70	7.30	2.2	4.1
11	8.64	10.21	3.0	4.4

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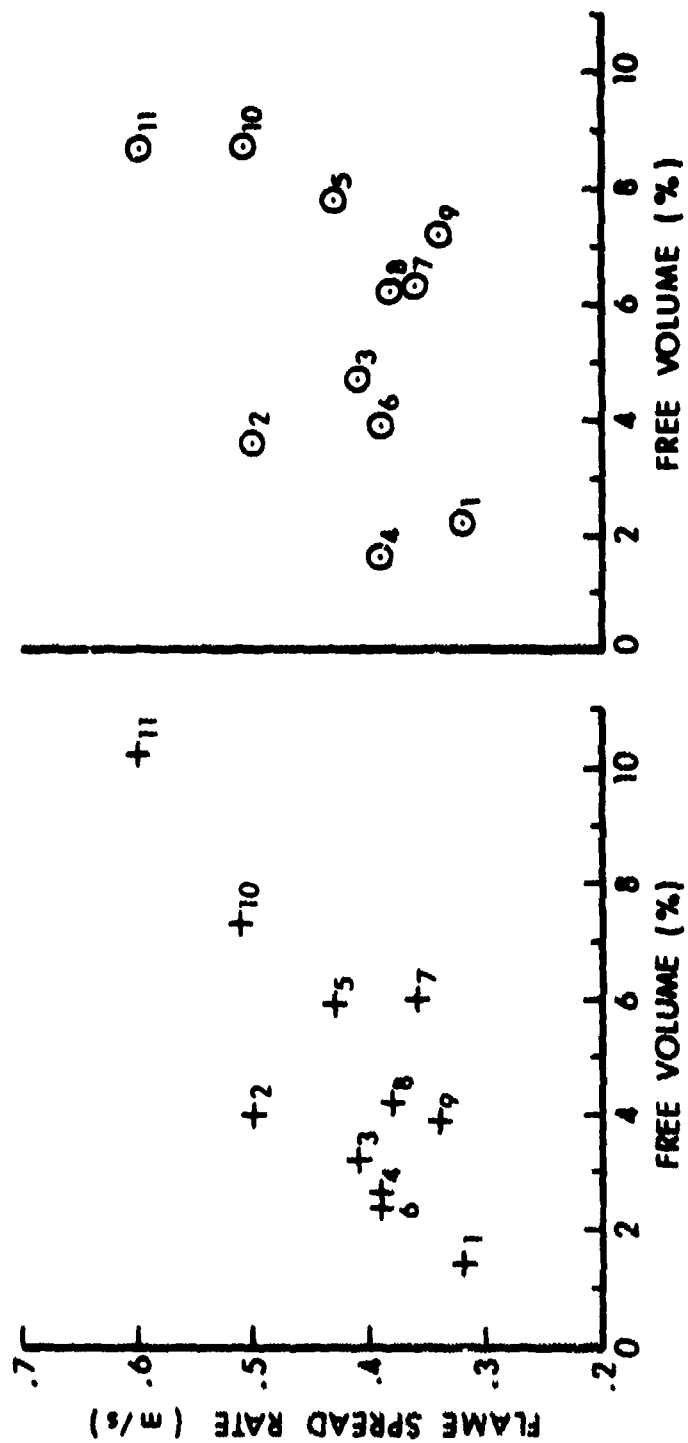
Figure 20. Relationship between flame spread rate and free volume. Subscripts refer to lot number: +, values calculated by equation (3) and ⊙, values calculated by equation (4).

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These values are listed in Table 4 and in Figure 20. Comparison of the free volumes obtained from equation (3) and (4) shows that in all cases, except lot 4 and lot 11, the total free volume including voids is larger than that accessible from outside of the grain. These volumes can be compared to the mercury intrusion values. One comparison is to relate the volume of mercury penetrating through passageways larger than 0.1 micron, a plateau in Figure 13, to the density derived values. In this case, the mercury values are smaller and this is reasonable as small diameter pores have not been penetrated. However, examining the smallest radii of 0.003 microns, the volumes are too large for lots 1 and 6. It is therefore concluded that the sharp rising asymptote occuring at the highest mercury pressures either elastically and/or inelastically constricts the black powder grain. For this reason, the mercury intrusion data is suspect beyond 10,000 psi or 70 kg cm^{-2}.

From the density measurements, both estimates of free volume are shown as a function of flame spread rate in Figure 20. The better correlation is given by the ether displacement volume reflecting the volume accessible from outside the grain.

If the volume occupied by moisture is considered, then it is clear that a small percentage of water could be significant in relation to the measured free volume of black powder. This could be the physical effect of water on burning rate. Shulman[25] and Plessinger[7] have measured the effect of water on burning rate and their data is reproduced in Figure 21. Since there could be a difference in equipment, these data sets should be examined independently, but in either case the trend is clear and it is suggested here, based on inference alone, that water plugs and occupies passageways to retard burning rate.

V. FUTURE PLANS

The suggestion is made that for future work a density range and volatile content for charcoal be established. Also, compaction pressure for making black powder should be selected and it is proposed that surface area and porosity measurements guide this study. In selecting compaction pressure, die dimensions should be considered and the density distribution determined. The density of black powder is an indirect measure of gross structure being sensitive to the density of charcoal and particle size of ingredients. From present work, it is proposed that the internal structure be measured and controlled. In such a study it is proposed to generate a standard meal and form black powder samples that provide a greater variation in density, porosity and surface area to better document the effect of these variables.
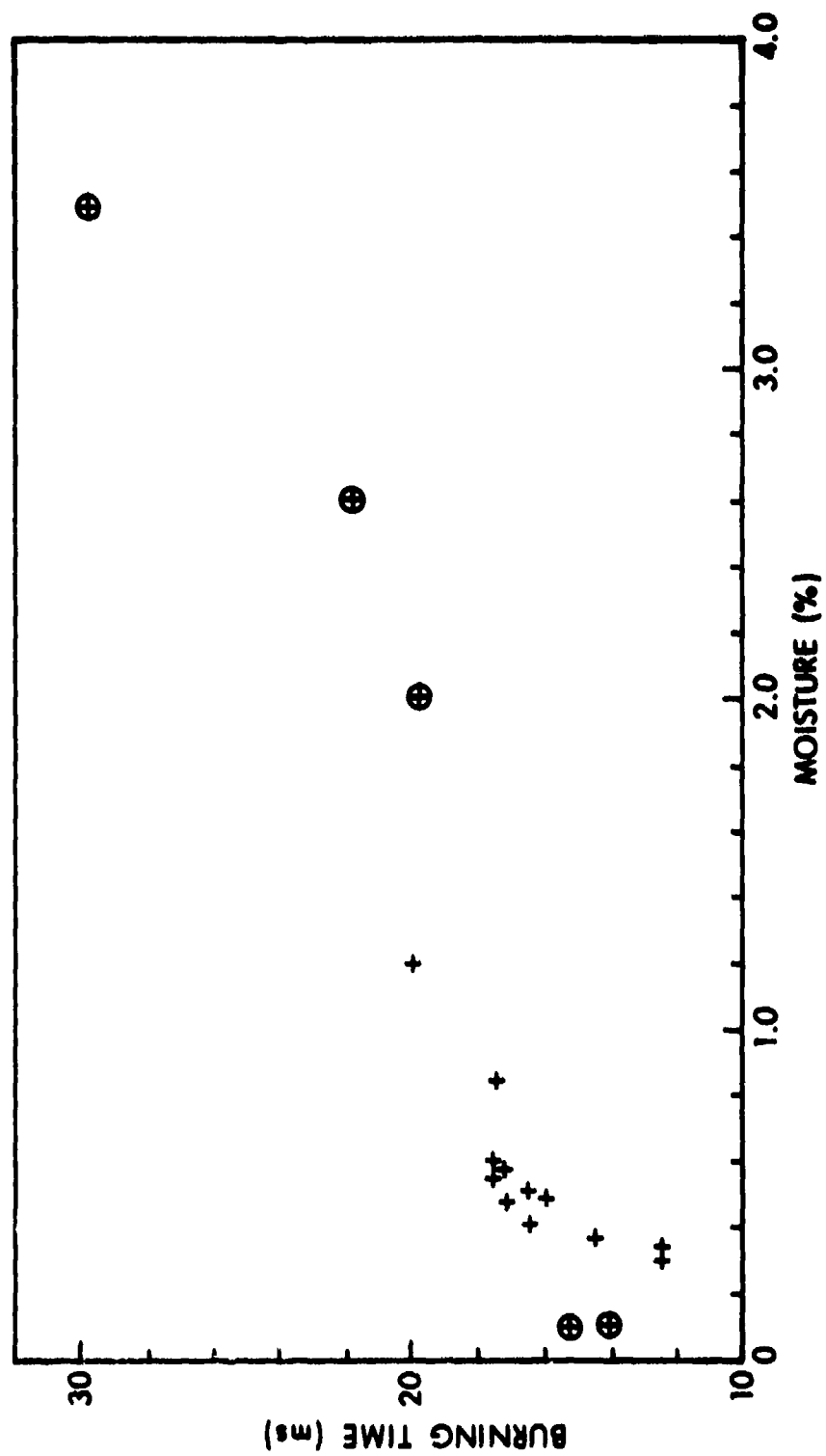
Figure 21. Relationship between burning time and moisture content

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VI. SUMMARY

1. It was found that compaction of black powder meal produces considerable plastic flow and forms a conglomerate matrix of interconnecting passageways to the extent that the internal free volume is but a few percent.

2. Correlations between internal surface area, pore volume, and density were made with burning rate.

3. It was suggested that the adverse effect of water is the result of occupying the free volume of black powder and retarding burning rate.

4. From the above relationships and S.E.M. microphotographs the hypothesis was established that an increase in the degree of openness of black powder grains increases burning rate.

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APPENDIX
PARTIAL TRANSLATION OF VIELLE ARTICLE-REFERENCE 30 PAGES 306-325
by Eli Freedman

II. First Series--Agglomeration of Pulverized Materials

We have used the materials of RWP brown powder. These materials, ground and sieved through a silk gauze, were agglomerated dry by compression with a hydraulic press, using the steel mold already described.

We thus obtained pastilles of 20-mm diameter, about 3.5 mm high; and blocks of triple weight, 10.5 mm high. Six pastilles or 2 blocks of the same density constituted a charge of loading density 0.6; 3 pastilles or 1 block permitted the carrying out of comparative experiments at a loading density of 0.3.

The sections of the pistons used for the measurement of pressure were configured in a way to furnish similar crushings so as to make the tracings comparable: a piston of 0.5 cm^2 for the density 0.6, and the one of 1.33 cm^2 for the density of 0.3.

The crushings were carried to 3.5 mm and 4 mm by the use of copper cylinders similar to the regulation cylinders, but with a partially-reduced cross section, ratio of similitude $1/\sqrt{2}$.

Table 11, gives the results of the tests (only one half of the table is reproduced here given as illustration).*

TABLE 11

| | | Duration of Combustion | |
Density	Thickness	Density of Loading 0.6	Density of Loading 0.3
1.922	10.44	98.07	125.0
1.785	11.19	87.13	108.80
1.760	11.47	48.33	74.57
1.724	11.65	17.36	28.78
1.635	12.09	5.22	12.01

The results of these 3 determinations are represented in Figure 15 (of text), whose abscissas represent the real densities of the pastilles, and the ordinates, the durations of combustion. [Curve reproduced in Sasse's text as Figure 2].*

Brackets enclose translator's comments or notes.

These four curves have a common shape. At low densities up to a value of about 1.720, the duration of combustion is effectively independent of thickness whether the combustion occurs at a charge density of 0.3 under a maximum pressure of 1200 kg, or at a charge density of 0.6 under a pressure reaching 3000 kg. However, combustion time slowly increases up to four times the combustion time of elementary dust; then, suddenly, between densities of 1.720 and 1.800, the influence of thickness suddenly manifests itself and the combustion duration increases with extreme rapidity, tending to the limits characteristic of the compact materials; i.e., effectively proportional to the thicknesses.

This effect of compression is hardly unique to brown powders. The same functioning is observed with black powder materials reduced to the powdered state.

V. General Mode of Action of Compression

In summary, for all of the materials that we have reviewed here, the influence of compression on the duration of combustion occurs in an identical way.

Four regimes of compression can be distinguished which correspond to four very different combustion modes.

In the first regime, with the weakest compression, the combustion duration of the materials does not differ from that of the elements which compose it *placed next to each other*. It is consequently independent of thickness.

In the second compression regime, the combustion duration progressively rises up to values reaching 4 or 5 times those that correspond to the grains of elementary dust, but this duration nevertheless remains independent of the thickness. *It is this type of material that normally constitutes the black or brown powders actually used by artillery.*

The third regime is characterized by an extraordinarily rapid variation of combustion duration with density; the influence of thickness appears and is accentuated more and more. This type of material is sometimes introduced during manufacture, when compression is required to achieve excessive slowing-up of the duration of combustion of the elementary grain; but the extreme irregularity of the products does not normally permit these types of materials to be used.

In the fourth regime, the materials, now virtually compact, show durations of combustion slowly increasing with density and proportional to their thicknesses.

This succession of phenomena can be very simply explained, based on the known laws of gas flows through capillary orifices. It is known that

.the discharge, Q, of fluids across capillary canals of all kinds is given by expressions of the type of Poisuille's law,

$$Q = KPD^{\frac{1}{4}}/L$$

where D = diameter; P, pressure; L, length; and K, a coefficent characteristic of the fluid; and that consequently, the flow velocities, proportional to Q/D^2, decrease with extreme rapidity with the dimension of the orifices.

It is conceivable that in the phenomenon of the penetration of incandescent [flaming] gas across a heterogeneous mass, the interstices of a certain order of magnitude, which will be called the first order, alone play an effective role; the orifices of lower order, e.g., 10 times less, permit the flame to propagate only with a velocity 100 times less.

In relation to the above ideas consider the material formed by agglomeration of grains at the lowest degree of compression. A system of interstices exists in the charge which assures flame spreading to all of the grains without a noticeable delay, or at least for a time duration negligible with respect to the combustion time of the grains themselves. This mode of combustion is made evident in the numerous examples given above, showing the identity of the durations of combustion of the free grain with the weakly-compressed blocks. This is the first phase of compression at work.

Under stronger compression the interstices are reduced by a sort of tangling of the grains, whose surfaces in contact become married in a more and more complete way while blocking or reducing to a very small dimension the entire grid of primitive interstices.

There results a new system of canals, whose dimensions remain of the original order; rather than limiting on the average each elementary grain, as in the primitive state, they limit a polyhedral kernel formed by the partial agglomeration of several grains; and it is readily conceivable that the average duration of combustion of these kernels can vary continuously with compression up to values double, triple, etc., the duration of combustion of the prim   .e grains without, however, a residual system of first-order canals ceasing to exist in the mass, thus assuring a combustion duration independent of the external dimension of the grain. This is the working of the second phase of compression.

Under increasing compression, the number of first-order canals is reduced enough so that the dimension of the kernels which they limit become of the order of a milled grain. One falls therefore into the irregularities which we have observed in the third phase of compression of the raw materials forming the grains at the moment where the influence of thickness is starting to be noticed. These irregularities continue

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until all of the first-order canals have disappeared and the influence of thickness definitely shows up.

The fourth phase of compression starts at this moment. The materials approach the limiting state corresponding to a perfect continuum and the velocities of combustion vary only very slowly with density. It is in fact conceivable that this velocity results from the conductibility of the materials for the heat accruing from *a slow velocity of infiltration of glowing gas across* the second-order interstices, whose dimensions are progressively reduced. From there on, there is a slow decrease of combustion velocity, while the density rises up to the practically realizible limits.

VI. Influence of Pressure on the Duration of Combustion

The results in Tables XI, XII, and XIII relating to the influence of compression on the mode of combustion of the pulverized raw materials permit the deduction of several qualitative conclusions about the influence that pressure exerts on combustion velocity.

If one considers materials of density greater than 1.780, for which the proportionality of the duration of combustion to thickness is effectively realized, i.e., the materials that function as compact, one obtains the following numbers [in the table] for the ratio of the durations of combustion of the same elements to the charge densities 0.3 and 0.6 (maximum pressures 3200 and 1300 kg).

The general average of these numbers is about 1.25, and the exponent of the pressure that takes account of this variation of the duration of combustion is about 0.25. This value was previously noted in Chap. II, as resulting from the comparison of the durations of combustion of compacted materials of 30/40 powder burning at densities 0.3 and 0.6.

When one considers materials of density less than 1.780, the ratio rises rapidly and attains values greater than 2.00. Whatever the errors which lead to irregularities in the ratios obtained in some of the pairs of experiments, we can certainly conclude that the influence of pressure increases significantly when the compactness of the materials diminishes. The ratio 2.5 effectively recapitulates the proportionality of the durations of combustion to pressures.

The discrepancy of the pressure exponents given by experimenters to represent the influence of pressure on the combustion velocity is easily explained by the variable nature of the raw materials on which they worked.

In any case, it is evident that this effect of pressure cannot be, *a priori*, regarded as identical for powders of different factories, and that it is important to determine it for each particular case.

(----page 325----)

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